United States Air Force
611th Civil Engineer Squadron

Elmendorf AFB, Alaska

Final

Addendum to the Sampling and Analysis Plan Galena Airport and Kalakaket Creek Radio Relay Station, Alaska

September 1994

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Prepared by:
Radian Corporation

September 1994

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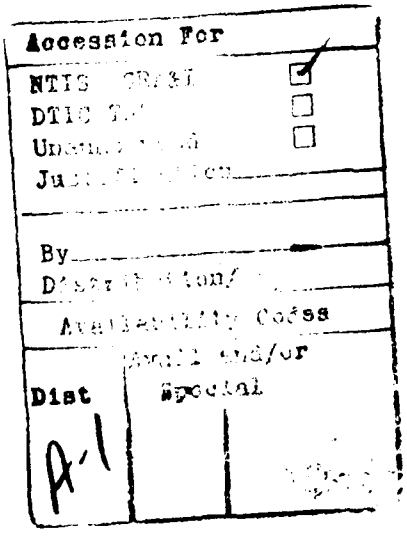

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Radian Corporation
P.O. Box 201088
8501 N. Mopac Blvd.
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13. ABSTRACT (Maximum 200 words)

This Sampling and Analysis Plan (SAP) describes the project objectives, the work to be performed toward the objective goals, and the methods used to obtain quality, interpretable data. The Quality Assurance Project Plan (QAPP) portion of this SAP defines analytical and QA/QC procedures; the Field Sampling Plan part of this SAP provides requirements and procedures for all field work to be conducted.

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QUALITY ASSURANCE PROJECT PLAN

The Sampling and Analysis Plan for the Remedial Investigation/Feasibility Study (RI/FS) at Galena Airport and for the Preliminary Assessment/Site Inspection (PA/SI) at Kalakaket Creek Radio Relay Station (RRS) consists of two sections, the Quality Assurance Project Plan (QAPP) and the Field Sampling Plan (FSP). Section 1.0, the QAPP, presents quality assurance (QA) information relevant to the sampling and analysis efforts at Galena Airport and Kalakaket Creek RRS, Alaska. This section was written following Air Force guidance found in *Handbook to Support the Installation Restoration Program (IRP) Statements of Work* (May 1991). As such, it is intended that this document present information required in a QAPP per the U.S. Environmental Protection Agency's (EPA) guidance *Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans* (EPA, 1980). Section 2.0, the FSP, provides the requirements and procedures for all field work to be conducted at Galena Airport and Kalakaket Creek RRS during the 1994 field season. This Sampling and Analysis Plan (SAP) has been updated, and is intended to replace the *1993 Sampling and Analysis Plan* (Radian, July 1993).

1.1 Introduction

A RI/FS is being conducted at Galena Airport and a PA/SI is being conducted at Kalakaket Creek RRS. Figure 1-1 shows the location of both installations along the Yukon River in interior Alaska. Within the framework of the Air Force IRP, the objective of the study being conducted at Galena Airport is to assess past hazardous waste disposal and spill sites and develop remedial actions consistent with the National Contingency Plan (NCP) for sites which pose a threat to human health and welfare or the environment. The objective of the PA/SI being conducted at Kalakaket Creek RRS is to confirm or deny the presence of contamination.

1.2 Project Definition

This section provides background information for Galena Airport and Kalakaket Creek RRS, and the investigations being conducted.

1.2.1 Installation Background

Galena Airport—Galena Airport occupies 166 acres and is located on a floodplain north of the Yukon River in interior Alaska. This deactivated Air Force installation, situated approximately 350 miles northwest of Anchorage and 280 miles west of Fairbanks, was the northernmost of the forward operating bases run by the USAF Pacific Air Forces (PACAF). Approximately 330 military personnel were assigned to the base when it was in operation. The base was deactivated during 1993. Currently, approximately 30 caretakers are present at the facility.

Galena Airport was constructed in 1941 by the Civil Aeronautics Authority (CAA, now the Federal Aviation Administration, FAA) as part of an overall airport construction program in Alaska. The U.S. Army acquired use of the airfield in 1943 and used it as an active refueling stop for aircraft bound for the Soviet Union under the Lend-Lease program. After World War II, the Army relinquished control of Galena Airport to the CAA, which operated the site as a civilian airport. Galena Airport served as a forward operating base for the Air Force from 1951 to 1993. During that time, fighter aircraft (most recently F-15s) were constantly on alert status as part of the Alaska Air Command air defense mission. The aircraft were supported by equipment and personnel stationed on the base.

Kalakaket Creek RRS—Kalakaket Creek RRS is located 22 miles southeast of Galena Airport, and encompasses approximately 316 acres of land. The RRS site is on the leveled top of a mountain at an elevation of 1950 ft above mean sea level.

Kalakaket Creek RRS became operational in May 1957 and provided network links between North River RRS, Bear Creek RRS, and Tatlina RRS. Microwave links to Galena Airport and Campion Air

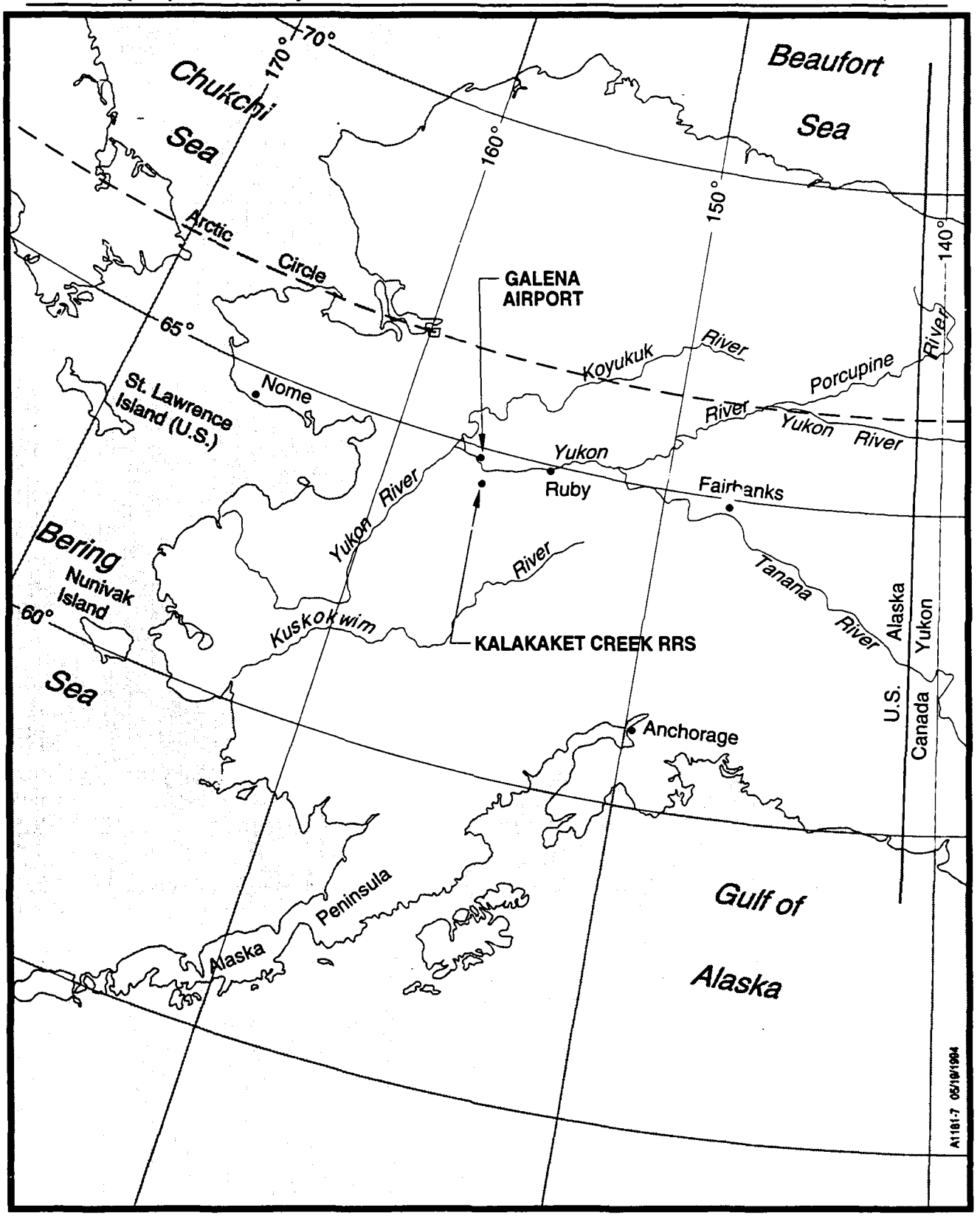

Figure 1-1. Location of Galena Airport and Kalakaket Creek RRS, Alaska

Force Station were added later, and the site was removed from service in 1973. Several structures were constructed on the site including an equipment/dormitory building, a vehicle maintenance shop, a paint storage building, a pair of diesel storage tanks, a pair of water storage tanks, four tropospheric scatter antennae, and two microwave antennae. All of these structures are still standing with exception of the paint storage building, which was partially demolished in 1984. In addition, a runway was constructed and accompanied by a temporary diesel storage tank, temporary vehicle garage, and aircraft support building. Asbestos containing material (ACM) was detected in the buildings on site.

In 1984, a site cleanup was performed and hazardous materials, contaminated soils, and salvageable equipment was removed from Kalakaket Creek RRS. Nonhazardous debris was buried in two pits located north of the eastern end of the runway and in one pit north of the RRS. Buried debris included CO_2 fire extinguisher cylinders, building materials from the demolition of the temporary vehicle garage and air services support shed located by the runway, and 3,250 cleaned, crushed drums.

1.2.2 Project Scope and Objectives

Galena Airport—The current environmental investigative work planned for Galena Airport encompasses sampling and testing activities at both newly defined areas and previously investigated sites. The overall objectives of the project are to:

- Conduct a round of groundwater sampling at all existing sites;
- Determine the presence or absence of dioxins at the Fire Protection Training Area (FPTA);
- Assess the impact of pesticide use on surface soils across the main base triangle;
- Characterize stockpiled soil contaminated with DDT for treatment; and
- Assess the impact of floodwater outfall on the soils near the Yukon River.

Kalakaket Creek RRS—An initial SI is planned at Kalakaket Creek RRS to identify Areas of Concern (AOC) and to confirm the presence or absence of contamination at the facility. Data from the investigation will be used to plan additional investigations, if required, or remove the potential AOCs from the list of possible sites recommended for further investigation. This will be accomplished by conducting a records search and collecting soil samples at potential AOCs for field screening and laboratory analysis.

The records search will be initiated prior to the start of field work. The specific goal of the search is to obtain recorded information on all potential AOCs and to determine past waste handling practices at the site. This information will focus the field screening activities on those areas that are most likely to contain soil contaminants and will help identify sources and types of any contamination. Also included in the records search task are plans to interview past workers at Kalakaket Creek RRS.

The goal of the field-screening task is to quickly and efficiently determine the presence or absence of contamination over larger areas while minimizing the total analytical cost. A subset of those samples collected and screened will be sent to an analytical laboratory for confirmational analysis.

1.2.3 Subcontractors and Their Roles

Two qualified subcontract laboratories were selected to perform sample analysis.

The subcontracted chemical analytical laboratories will perform the analytical work in accordance with this Sampling and Analysis Plan. Superior Laboratories, located in Martinez, CA, has been subcontracted to analyze water samples by the Alaska State Methods AK101 and AK102 (Appendix A). Radian Analytical Services will perform all other soil and water analyses.

Although not a subcontracted company, Radian will coordinate with the 611th CES to provide the following:

- Drums to collect soil cuttings and purge water;
- Surveying crew and surveying equipment to survey all sampling locations on a schedule communicated by the contractor; and
- Drum storage and treatment or disposal of Investigational Derived Wastes (IDW).

1.3 Project Organization and Responsibility

The organization of the RI/FS project team is presented in the following sections. A brief review of the primary staff and their responsibilities is given below.

1.3.1 Project Management

- Mr. Nelson Lund is the contract program manager, with responsibility for the contractual aspects of the project.
- Mr. Mike Green is the project manager, with responsibility for directing project planning activities and ensuring that qualified technical staff are assigned to the tasks. He also is responsible for overall technical quality and consistency of all project activities and deliverables.

The contract program manager and the project manager have overall responsibility to ensure that all activities are performed in accordance with United States Environmental Protection Agency (US EPA), USAF, state, and local requirements.

1.3.2 Quality Assurance

- Mr. Dan Anderson, the independent project QA officer, is responsible for planning, implementing, and tracking quality assurance activities and maintaining communication with quality control and analytical task staff members. His duties include ensuring that quality control data evaluation, data validation, and reporting procedures are followed and updating the QAPP as necessary. The ultimate goal of these activities is to produce data that satisfy the QA objectives for the program.

1.3.3 Health and Safety

- Ms. Stephanie Taylor is responsible for general Health and Safety Plan development and training for field personnel. She is also responsible for ensuring that the health and safety procedures are understood and followed by all field personnel through training and for reporting and correcting any violations of these procedures.

1.3.4 Analytical Services

- Ms. Jane Lindsey is the laboratory client services coordinator, with responsibility for the logistical aspects of sample analysis and reporting. These responsibilities include scheduling sample analysis, coordinating sample shipment, and issuing analytical results to the project staff.
- Ms. Debra Bisson is the project chemist in charge of coordinating sampling materials for the field teams, interfacing with the client services coordinator on laboratory problems, and reviewing analytical results.

1.3.5 Primary Task Leaders

- Mr. Todd Council and Ms. Becky Coel are responsible for the overall direction of the field investigation, including sampling report preparation.
- Ms. Sandy Smith is responsible for coordinating tasks relevant to constructing a baseline risk assessment for each site. This includes reporting and supporting the numerical risk estimates.
- Ms. Xandria Eykel is responsible for direction of tasks relating to the Community Relations Plan. This includes producing a photo notebook, interfacing with Air Force and Galena public officials, and helping the Air Force conduct public meetings at Galena.

As the work plans are developed, project team members will be identified based on their experience and ability to perform the required work. Resumes of all project personnel are available for review.

The task leaders are responsible for reviewing and updating training files for team members, and conducting training or refresher courses for all members to ensure that all field, QA, and Health and Safety procedures are understood and will be followed in the field. The team members are responsible for reading and understanding the protocols established in the SAP and Work Plan; the task leaders and the project director will provide time for review and be available to answer questions that may arise during that review.

1.4 Quality Assurance Objectives for Measurement Data

The purpose of a quality assurance/quality control (QA/QC) program is to produce data of known quality that satisfy the project objectives and that meet or exceed the standard requirements for the analytical methods. The QA/QC program will:

- Provide a mechanism for ongoing control and evaluation of measurement data quality; and
- Provide measures of data quality in terms of accuracy, precision, completeness, representativeness, and comparability to assess whether the data meet the project objectives and can be used for their intended purpose.

The basis for assessing precision, accuracy, completeness, representativeness, and comparability is discussed in the following subsections. Specific calculations for data quality measurements, and the data assessment procedure, are presented in section 1.9.3.

1.4.1 Definition of Criteria

This section defines the terminology associated with Quality Assurance Objectives (QAO) criteria to be used during the Galena Airport project.

1.4.1.1 Precision

Precision measures the reproducibility of repetitive measurements and is usually expressed in terms of imprecision. It is strictly defined as the degree of mutual agreement among independent measurements as the result of repeated application of the same process under similar conditions. Analytical precision is a measurement of the variability associated with duplicate (two) or replicate (more than two) analyses of the same sample in the laboratory and is determined by analysis of matrix spike duplicates or laboratory duplicates. Total precision is a measurement of the variability associated with the entire sampling and analysis process. It is determined by analysis of duplicate or replicate field samples and includes all possible sources of variability. Imprecision will be estimated for the Galena Airport project using the RPDs between duplicate measurements of laboratory control samples.

Precision goals are presented in tables in section 1.10 for each method and matrix. Precision goals will be met if all duplicate analyses of laboratory control samples agree within relative percent differences (RPD) specified in section 1.10. RPDs for laboratory control samples outside specified criteria indicate the analytical system is out of control and require samples to be reanalyzed. Precision will not be assessed by matrix spike duplicates nor field duplicates, both of which contain matrix effects which cannot be controlled. Results of these duplicate measurements will be used to evaluate the total imprecision possible in natural-matrix sample results.

1.4.1.2 Accuracy

Accuracy is a statistical measurement of correctness, and includes components of random error (variability due to imprecision) and systematic error (bias). It therefore reflects the total error associated with a measurement. A measurement is accurate when the value reported does not differ from the true value, or known concentration of the spike or standard. Analytical accuracy is typically measured by determining the percent recovery of known target analytes that are spiked into a field sample (a matrix spike) or reagent water or soil (a method spike) before extraction, at known concentrations. Surrogate

compound recovery is another spiking technique used to assess method accuracy for each sample analyzed for volatile and semivolatile compounds. The stated accuracy objectives apply to spiking levels at five times the method detection limits or higher. The individual methods provide equations for acceptance criteria at lower spiking levels.

Both accuracy and precision are calculated for specific sampling or analytical batches, and the associated sample results must be interpreted considering these specific measures. Application of calculated precision and accuracy to measurement sample results is discussed in section 1.13. An additional consideration in applying accuracy and precision is the concentration level of the samples. A procedure capable of producing the same value within 50% would be considered precise for low-level (near the detection limit) analyses of minor constituents, such as metals in groundwater samples, but would be unacceptable, and possibly useless, for major constituents at high concentrations.

Accuracy goals for laboratory control samples are presented in tables in section 1.10. Accuracy goals will be met if all individual laboratory control sample recoveries are within listed criteria. Laboratory control sample recoveries outside criteria indicate the analytical system is out of control and require samples to be reanalyzed.

1.4.1.3 Completeness

Completeness is calculated from the aggregation of data for all methods for any particular sampling event. For each method for each site, the number of valid results divided by the number of individual analyte results initially planned for, expressed as a percentage, determines the completeness for the data set. The objective for completeness is 90 percent. If any samples cannot be analyzed (e.g., holding time violations in which resampling and reanalysis were not possible, samples spilled or broken, etc.), the numerator of this calculation becomes the number of valid results minus the number of possible results not reported.

Valid results used to meet completeness objectives are those results which provide defensible estimates of the true concentration of an analyte in a sample. These valid results include data which are not qualified and data which QC results indicate qualification is necessary but the data may still be used to meet project objectives. Invalid results are those data for which there is an indication that the prescribed sampling or analytical protocol was not followed.

1.4.1.4 Representativeness

Objectives for representativeness will be defined for each sampling and analysis task and will be a function of the investigative objectives. Representativeness will be achieved, in part, through use of the standard sampling and analytical procedures described in this SAP. Representativeness is also determined or influenced by appropriate program design, considering elements such as proper well locations, drilling and installation procedures, and sampling locations.

1.4.1.5 Comparability

Comparability is the confidence with which one data set can be compared to other data. One objective for this QA/QC program is to produce data with the greatest degree of comparability possible. The number of matrices that will be sampled and the range of field conditions encountered must be considered in ultimately determining comparability. Comparability will be achieved by using standard methods for sampling and analysis, reporting data in standard units, and using standard and comprehensive reporting formats. Analysis of reference samples may also be used to assess comparability of analytical data produced by the laboratories.

1.4.1.6 Quality Objectives for the Site Characterization Data

Site characterization data for Galena Airport will be obtained from the collection and analysis of groundwater and soil samples during the RI/FS. While it is more difficult to measure the precision, accuracy, completeness, representativeness, and comparability of the data collected from these activities, several steps will be undertaken to ensure the data

obtained are representative, standardized, and as accurate as possible.

The compilation of hydrologic data will be conducted in several parts of the RI/FS site characterization and hydrogeologic assessment. Hydrologic data to be compiled will consist primarily of water-level measurements collected over specific time intervals. Electronic water level meters, or E-lines will have a unique identification number that can be used to document instrument use and performance. If more than one sounder is used during a sounding event, sounders will be checked for comparability of measurements.

Additional hydrologic data which may be collected during the RI/FS are pump discharge rates measured during well purging. The methods used to measure pump discharge rates will be determined by the uses of the discharge data. For monitoring well purging, discharge data are needed to demonstrate that the well to be sampled was adequately purged and that the discharge was sufficient to induce groundwater flow from the formation into the well casing. Discharge measurements to assure purging can be obtained with sufficient accuracy by using a bucket of known volume and a stop watch.

The Kalakaket Creek project goal for the PA/SI analytical data is to determine the presence or absence of contamination at the site. The analytical data, documentation, and reports must be defensible (the ability to withstand any reasonable challenge related to veracity or truthfulness) and verifiable (the ability to prove or substantiate any result or claim related to the documented record).

1.4.2 Goals

The QA objective (i.e., goal) for the Galena Airport and Kalakaket Creek RRS projects is to have all analyses performed with an analytical system that is in statistical control and meets method specifications. Numerically, the goal is to have all individual results traceable to a laboratory control sample whose recovery (for both precision and accuracy) is within method-specified limits. Method specifications will be used as tolerance limits for the project. Laboratory-derived limits used to statistically monitor analytical system control will be within method specifications. The method-specified limits for laboratory control samples are supplied in section 1.10 along with method-specified limits for spike recoveries in natural matrix samples. Inaccurate or imprecise recovery of laboratory control samples *will* invalidate results; inaccurate or imprecise recovery of spikes in natural-matrix samples *will not* invalidate results. Poor recoveries of spikes in natural-matrix samples indicates the potential for matrix effects. A conclusion of matrix effects must be supported by laboratory control sample results within acceptance criteria for the analytical batch for which the matrix spike was performed.

1.5 Sampling Procedures

This section describes components of the sampling procedures that will be performed to meet the QA objectives for the Galena Airport RI/FS and Kalakaket Creek RRS PA/SI.

1.5.1 Sampling Protocols

Detailed sampling protocols are provided and discussed in the FSP (section 2.0 of this document). Prior to the beginning of the sampling event, a detailed sampling plan will be prepared and the field task leaders will meet with the assigned sampling personnel and review the purpose and objectives of the event. This meeting will provide final clarification of the sampling event details. Topics of review and discussion will include sampling locations, types of samples to be collected, number of samples collected, sample numbering, preservation requirements, parameter(s) to be analyzed, sampling procedures, equipment decontamination procedures, and chain-of-custody requirements.

1.5.2 Sample Handling

The field manager is responsible for ensuring that samples are collected with properly decontaminated equipment and containerized in properly cleaned sample bottles. A summary of the recommended sample containers, volume, preservation, and hold times for each analytical method and sample matrix is provided in Table 1.5-1.

Table 1.5-1
Water and Soil Sample Storage and Preservation Requirements

Parameter	Analysis Method(s)	Holding Time[a]	Container(s)[a,b]	Preservation	Storage Requirements
ORGANIC COMPOUNDS:					
Organochlorine Pesticides and PCBs	SW8080	7 days until extraction, 40 days to analyze (w); 14 days until extraction, 40 days to analyze (s)	One 1-Liter glass bottle with Teflon® seals (w); 8-oz wide-mouth bottle with Teflon® liner (s)	None (w, s)	4°C
Volatile Organic Compounds	SW8240/ SW8260	14 days (s)	Three 40-mL glass vials with Teflon® seals (w)	HCl to pH <2 (w); none (s)	4°C
Semivolatile Organic Compounds	SW8270	7 days until extraction, 40 days to analyze (w); 14 days to extraction, 40 days to analyze (s)	One 1-Liter glass bottles with Teflon® seals (w); 8-oz wide-mouth glass bottle with Teflon® liner(s)	None (w, s)	4°C
Polychlorinated Dioxins and Furans	SW8280	30 days until extraction, 45 days from collection date to analyze	8-oz wide-mouth glass bottle with Teflon® lined cap (s)	None (w, s)	4°C
Diesel Range Organics (DRO)	AK102.0	7 days until extraction, 40 days to analyze (w); 14 days to extraction, 40 days to analyze (s)	One 1-Liter amber glass bottles with Teflon® seals (w); 8-oz glass wide-mouth bottle with Teflon® liner (s)	HCl pH <2 (w); none (s)	4°C
Gasoline Range Organics (GRO)	AK101.0	14 days to analyze (w, s)	Three 40-mL amber or clear (protect from light) glass vials with Teflon® seals (w); 4-oz wide-mouth glass bottle with Teflon® liner (s)	200 µL of 50% HCl (w); none (s)	4°C

Table 1.5-1
(Continued)

Parameter	Analysis Method(s)	Holding Time[a]	Container(s)[a,b]	Preservation	Storage Requirements
INDICATOR AND INORGANIC COMPOUNDS/PARAMETERS:					
Alkalinity	SM403	Field Test	Glass beaker	None	Analyze immediately
Specific Conductance	SW 9050	Field Test	Glass beaker	None	Analyze immediately
pH	SW9040 (w) SW9045 (s)	Field Test	Glass beaker	None	None, analyze immediately
Temperature	E170.1	Field Test	250-mL polyethylene bottle	None	Analyze immediately
Moisture	A-D2216	Not specified	Glass beaker	None	4°C
Arsenic	SW7060	180 days (w)	500-mL polyethylene bottle (w)	pH <2 with HNO_3 (w)	4°C
Lead	SW7421	180 days (w)	500-mL polyethylene bottle (w)	pH <2 with HNO_3 (w)	4°C
ICP Metals	SW6010	180 days (w, s)	500-mL polyethylene bottle (w); 8-oz wide-mouth glass bottle with Teflon®-lined cap (s)	pH <2 with HNO_3 (w); none (s)	4°C

[a](w) = water; (s) = soil

[b]All containers are pretreated and cleaned before being purchased by the laboratory. Sample container size is approximate and may be changed as appropriate to address specific field and laboratory conditions.

E = U.S. Environmental Protection Agency
SW = *SW846 Third edition*
SM = Standard Methods for the Examination of Water and Wastewater, 16th edition
A = ASTM

1.5.3 Sampling Equipment Decontamination

Equipment decontamination is an integral part of the data collection and QA process. The implementation of proper decontamination practices and procedures will begin in the field prior to use of sample collection equipment. All nondedicated field sampling equipment will be decontaminated before use and after each sample location, according to procedures outlined in the Field Sampling Plan. Wash water and other fluids generated during decontamination will be put in containers and clearly labeled. The drums will be moved to an Air Force specified storage area. The IDW will be treated and/or disposed by the Air Force following the guidelines in the IDW memorandum dated 24 September 1993 (Radian, 1993).

1.6 Sample Custody

Sample possession during all sampling efforts must be traceable from the time of collection until the results are verified and reported. The sample custody procedures provide a mechanism for documentation of all information related to sample collection and handling.

The field sampling task leader will be responsible for ensuring that the field team adheres to proper custody and documentation procedures for all sampling operations. Preformatted chain-of-custody forms will be used as the primary documentation mechanism to ensure that information pertaining to each sample is recorded. In addition, field notebooks and a master sample logbook will be maintained for all samples collected. Copies of the chain-of-custody forms and the field logs will be retained in the project file.

1.6.1 Field Operations

This section describes field procedures for maintaining sample custody. Other information describing field operations may be found in section 2.2 of the Field Sampling Plan.

1.6.1.1 Field Records

Field personnel will be required to keep accurate written records of their daily activities in a bound logbook. All entries will be legible, written in waterproof ink, and contain accurate and inclusive documentation of an individual's field activities, including field data and observations, any problems encountered, and actions taken to solve the problem. The type of data recorded in the field logbook includes field measurements (pH, conductivity), ambient conditions, and any other information pertinent to sample collection. Entry errors or changes will be crossed out with a single line, dated, and initialed by the person making the correction. Since entries will be made by several individuals over the coarse of the investigation, each person must sign and date their entries. Field logbooks will be available for review by the QA coordinator during systems audits or at any other time for QC checks by field team leaders. This documentation provides verification of sampling procedures.

1.6.1.2 Sample Labels

Each sample collected will receive a sample label. Sample labels will identify the sample by documenting the unique sample identification number, the sample type, analytical method, the sampler's initials, date collected, and the preservation method used. Sample labels are waterproof and will be completed with a permanent marker and affixed to the sample container, and protected with clear tape.

1.6.1.3 Sample Identification

A numbering system will be developed to uniquely identify all samples. This numbering system will provide a tracking procedure to allow data retrieval and will ensure that sample identifiers are not duplicated. A listing of the sample identification numbers will be maintained by the project data administrator, and the field supervisor will ensure that it is universally applied to samples collected during this project.

1.6.1.4 Chain-Of-Custody

All sample shipments will be accompanied by the chain-of-custody record. The original record plus copies will accompany the shipment with one copy retained in the project file. Another copy will be returned to the project teams with analytical results and the original returned when the sample is disposed. An example of a chain-of-custody form is shown in section 2.0.

When appropriate, as in the case of overnight shipment, the custody record should contain a statement that the samples were delivered to the designated location and the date and time noted. Sample collection and shipment will be coordinated to ensure that the receiving laboratory has staff available to process the samples according to method specifications.

All shipping containers will be secured with chain-of-custody seals for transportation to the laboratory. All samples will be sent out on chartered or commercial flights with Larry's Air Service or Frontier Flying Service to Fairbanks, at which point they will be transferred to Federal Express for overnight delivery to the laboratory.

1.6.1.5 Shipping Procedures

The objective of sample handling procedures is to ensure that samples arrive at the laboratory intact, at the proper temperature, and free of external contamination. All samples will be shipped to the analytical services laboratory via overnight carriers, according to Department of Transportation standards. Chain-of-custody procedures will be followed during transport.

Sample packaging requirements for hazardous materials requiring interstate transport are defined in the *Code of Federal Regulations* 40 (CFR) 49, Chapter 1, Part 171. These requirements outline in detail the proper classification and procedures for transportation of hazardous materials; however, it is not anticipated that any of the samples will be classified as hazardous.

Generous amounts of double-bagged ice will be packed with the samples to maintain the temperature at 4 °C or less. The double-bagged ice will be of sufficient volume and will be distributed in the coolers so that the temperature can be maintained for two days. When the samples are delivered to the laboratory, the temperature of each cooler will be measured and recorded on the chain-of-custody form or addendum. The samples will be immediately placed in the sample control refrigerator after sample log-in.

The following procedures will be used to prevent bottle breakage and cross-contamination:

- All samples will be transported inside hard plastic coolers.
- All 40 mL volatile organic analysis (VOA) bottles will be placed in blocks of foam.
- All other glass bottles will be placed in plastic mesh sleeves to prevent glass-to-glass contact.
- Plastic sample bottles, bags of ice, and protective foam blocks will be used to separate glass bottles.
- Vermiculite or absorbent paper will be used to isolate the bottles from each other.
- The coolers will be taped shut and sealed with chain-of-custody tape to indicate unauthorized opening of the cooler.
- Samples that are known or suspected to be highly contaminated (based on field screening data or observation) will be packaged and shipped separately from other samples. Field screening will be noted on the accompanying chain-of-custody.

1.6.2 Laboratory Operations

The analytical services laboratory will follow standard operating procedures (SOPs) for handling, identification, control, and chain-of-custody procedures and to maintain the validity of the samples. These SOPs are based on the use of a laboratory information management system (LIMS) for tracking samples from receipt through reporting of the analytical results.

1.6.2.1 Sample Handling

The following section describes the activities related to sample receipt, storage and tracking.

Sample Receipt

At a minimum, sample receipt considerations will address the following:

- Upon receipt, the sample custodian will inspect all sample containers for integrity. The presence of leaking or broken containers or custody seals will be noted on the chain-of-custody form. The sample custodian will sign the chain-of-custody form (with date and time of receipt), thus assuming custody of the samples.

- The information on the chain-of-custody form will be compared with that on the sample tags and labels to verify sample identity. Any inconsistencies will be resolved with the field sampling representative before sample analysis proceeds.

- The temperature of incoming coolers of samples will be checked and the temperature recorded on the internal chain-of-custody.

- Preserved samples (i.e., those requiring pH adjustment), except for VOC samples, will be checked and any improperly preserved samples noted on the chain-of-custody.

- Samples will be moved to a locked sample storage refrigerator for storage prior to analysis. A separate storage refrigerator will be used to store low level samples for volatile organic analysis. The storage location will be recorded on the chain-of-custody record.

- The sample custodian will retain the original chain-of-custody form and provide copies to each laboratory section manager and one to the main sample log maintained at the laboratory.

- The sample custodian will alert the appropriate section managers and analysts of any analyses requiring immediate attention because of short holding times.

Sample Storage

Samples will be placed in locked storage refrigerators prior to sample preparation and analysis. The storage refrigerators are located in sample control and are maintained at 4° ± 2°C. Analytical laboratory personnel will request or check out samples for analysis from the sample custodian. The sample custodian and analyst will sign and date the internal chain-of-custody record to acknowledge transfer of custody to the analyst.

If samples are known or suspected to be highly contaminated, laboratory sample control personnel will be notified so those samples can be stored separately from less contaminated samples, minimizing the potential for cross contamination.

Sample Tracking

Organic Analysis—For samples that require extraction prior to analysis, a sample extraction form will be completed during the time of extraction. When samples are extracted for analysis by GC, GC/MS, or liquid chromatography, all pertinent data will be entered on the sample extraction form and/or recorded in a bound laboratory notebook. Extraction data are entered into the laboratory information management system by the person performing the extraction. A hard copy of the form will be printed and used as the vehicle for custody transfer to the analyst. Copies will be provided to the analysts to inform them that extracts are ready for analysis. The bound laboratory notebook will be kept in the extraction laboratory.

Extracts will be maintained in refrigerated storage by the sample preparation section until transferred to the analysts.

Metals Analysis—Samples will be received by the inorganic sample preparation section for digestion prior to analysis for metals. When samples are prepared for digestion, the preparation technician will fill out a sample digestion record.

All information regarding sample digestion will be entered onto the sample digestion record and recorded in a bound laboratory notebook as the sample preparation proceeds. The digestion record will be maintained to acknowledge custody transfer of digestates to the metals analysis section. Upon completion of sample digestion, a copy of the sample

digestion record will be provided to the metals analysis section to alert them that digestates are ready for analysis. The bound laboratory notebook containing the digestion record will be retained by the metals digestion laboratory.

1.6.2.2 Sample Identification

As samples are logged into the laboratory sample tracking system [Sampling and Analysis Management system (SAM®)] each sample is assigned a unique sample control number. This number is derived from the date of receipt and the sample receiving area involved, and is correlated with the field sample numbers obtained from the field chain-of-custody forms as both numbers are entered into the SAM® system for a given job. Analytical requirements for each sample are entered into the computer. A hard copy of the work order and other information is printed and filed with the received documentation in the document control center. Labels are printed with sample information and secured to each sample. Data sheets and work sheets are printed for each batch of samples and are distributed to the appropriate laboratory managers. The work sheets list sample information, storage location, and analytical requirements.

1.6.2.3 Sample Custody Records

Sample custody and documentation in the Radian Analytical Services Laboratory are organized around SAM®. SAM® is a computer software system specifically designed for tracking and handling the large amount of information required for the efficient management of an analytical chemistry laboratory.

Following sample log-in, the samples are placed in a designated locked storage area. Samples are maintained at 4°C (±2°C) from the time of receipt until the analyses are complete. Subsequent sample custody and all transactions are documented. Samples are checked in and out of the sample control area in a bound notebook.

The analyst receives the samples from the Sample Control Center and completes the sample work sheets or custody sheet. After analysis, the sample is returned to the designated storage location in the Sample Control Center. The sample is stored until the assigned time, or until written permission is given to either properly dispose of or return the sample to the client. All sample documentation is maintained in Document Control, a restricted access area.

1.6.2.4 Record Keeping

Data related to sample preparation and analysis procedures and observations by laboratory analysts will be recorded in bound and numbered laboratory notebooks issued by the laboratory document control section. Laboratory notebook pages will be signed and dated daily by laboratory analysts. Corrections to notebook entries will be performed by drawing a single line through the erroneous entry and by writing the correct entry next to or above the one crossed out. All corrections will be initialed and dated by the analyst.

1.7 Calibration Procedures and Frequency for Field Test Equipment

Calibration procedures for field and laboratory instrumentation are performed to ensure that the instruments are operating properly and produce data that can satisfy the objectives of the sampling program.

The analytical and health and safety screening instruments that may be used in the field during the RI/FS are listed below:

- Organic vapor meter (OVM);
- Specific conductance meter;
- pH meter;
- Thermometer;
- Infrared analyzer;
- Soil test kit for PCBs; and
- Soil test kit for DDT.

The instruments will be calibrated according to manufacturers' specifications before and after each

field use, or as otherwise described below. Instruments will be calibrated, at minimum, each day during field use.

1.7.1 Organic Vapor Meter (OVM)

An organic vapor meter (OVM) contains an internal microprocessor which performs the necessary calculations for the calibration process. The manufacturer recommends using a span gas and a zero gas to calibrate the instrument. The recommended span gas is isobutylene at 100 ppmV in air, and clean ambient air is the zeroing gas. A commercial source of zeroing gas should be used if any doubts about the purity of the ambient air exists. After the instrument has been exposed to the zeroing gas and the span gas, the microprocessor computes and displays a message of either successful calibration or error in calibrating; if the latter message is shown, the instrument is recalibrated. If the error message continues, the instrument is sent for repair and a backup instrument is used.

1.7.2 Specific Conductance Meter

An Omega Model pH-60 specific conductance meter will be used to measure the conductivity of surface water, groundwater, and wastewater systems. The Omega Model pH-60 meter is a battery-operated combination pH and conductivity meter with automatic temperature compensation. This instrument will be calibrated daily according to the manufacturer's specifications. This electronic calibration will be conducted by zeroing the meter and then calibrating with a standard potassium chloride (KCl) solution.

1.7.3 pH Meter

An Omega Model pH-60 meter will be used for determining pH (to $\pm$ 0.1 pH unit) of surface water, groundwater, and wastewater systems. This instrument will be calibrated daily according to the manufacturer's specifications with a multipoint calibration prior to sampling activities, and with a single-point check at each well location. If the drift exceeds 0.2 pH units, a new multipoint calibration will be performed.

1.7.4 Temperature

On-site water temperature is measured using a factory-calibrated standard mercury-in-glass thermometer.

1.7.5 Total Recoverable Petroleum Hydrocarbons

A General Analysis Corporation TPH field analyzer will be used to determine total recoverable petroleum hydrocarbons (TRPH) in water and soil field screening samples. The analyzer utilizes an infrared detector that is factory calibrated with EPA Method 418.1 reference oils. Two reference standards (40 ppm TRPH and 400 ppm TRPH) will be analyzed each day prior to sample analyses, after every ten samples, and at the end of a batch to insure proper instrument performance. The analyses of these standards must be within $\pm$20% of the true value. If analyses of a second set of standards is still outside this range, then the instrument is to be recalibrated.

1.7.6 Electronic Water Level Meter

Water level measurements will be obtained using a factory calibrated electronic water level meter and will be measured to the nearest 0.01 ft.

1.7.7 PCB Screening by Immunoassay

Soil samples from Galena and Kalakaket Creek RRS will be analyzed for PCBs in the field laboratory using an enzyme immunoassay test kit. In this method (Appendix A), samples are initially prepared by mixing a measured amount of soil with methanol to extract the PCBs. The mixture is filtered, diluted and added to a specially coated tube. PCBs are retained on the solid phase of the coated tube and the rest of the sample is washed away. A PCB-enzyme conjugate is then added to allow binding to the antibodies. Unbound conjugate is washed away and the amount retained is inversely proportional to the amount of bound PCB. Enzyme substrate and chromogen are added to the tubes for color development by the bound enzyme. The intensity of the color is inversely proportional to the amount of bound PCB.

1.7.8 DDT Screening by Immunoassay

Soil samples from Kalakaket Creek will be analyzed for DDT and its metabolites in the field laboratory using an enzyme immunoassay test kit. In this method (Appendix A), samples are initially

prepared by mixing a measured amount of soil with methanol to extract DDT and its metabolites. The mixture is filtered, diluted, and added to a specially coated tube. DDT and its metabolites are retained on the solid phase of the coated tube and the rest of the sample is washed away. A DDT-enzyme conjugate is then added to allow binding to the antibodies. Unbound conjugate is washed away and the amount retained is inversely proportional to the amount of bound DDT. Enzyme substrate and chromogen are added to the tubes for color development by the bound enzyme. The intensity of the color is inversely proportional to the amount of bound DDT.

1.8 Analytical Procedures

This section contains brief descriptions of calibration procedures and analytical methodology for the analysis of water and soil samples that will be collected during various phases of the RI/FS. In this section, the analogous water and soil methods are described together and detection limits are tabulated for each method.

1.8.1 Identification of Methods

Methods to be used for sample analysis are presented in Table 1.8-1. Most of the laboratory methods identified in this document were published by EPA in *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods SW846*, Third Edition, U.S. EPA, 1986, or *Methods for Chemical Analysis of Water and Wastes* (U.S. EPA, 1983). Additional methods identified were published in *Criteria for Identification of Hazardous and Extremely Hazardous Wastes," "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act*, 40 CFR, Part 136, *Federal Register* 49 (209), 26 October 1984, *Annual Book of ASTM (American Society for Testing and Materials) Standards*, Volume 4.08, and *Standard Methods for the Examination of Water and Wastewater*. The Alaska methods AK101 and AK102 are included in Appendix A.

1.8.2 Method Detection and Quantitation Limits

This section presents and defines limits to be used in describing detectable concentrations.

1.8.2.1 Terminology

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.

1.8.2.2 Procedures

Radian performs MDL studies on an annual or quarterly basis (depending on the method) to demonstrate that it can meet or exceed the method recommended MDLs. The U.S. EPA procedure used for establishing MDLs is described in Appendix B to Part 136 *Definition and Procedure for the Determination of the Method Detection Limit* - Revision 1.11, 40 CFR 136, 1986. This procedure consists of analyzing (using all sample processing steps specified in the method) seven aliquots of a standard spiked at three to five times the expected MDL. The MDL is defined as three times the standard deviation of the mean value for the seven analyses. In addition, the laboratory may establish reporting limits which are verified by the MDL studies and included on the laboratory's analytical reports.

1.8.2.3 Values

Analytical methods and corresponding reporting limits are presented in Tables 1.8-2 to 1.8-8.

1.8.3 Method Description and Calibration

This section describes the extraction and analytical methods to be used during the RI/FS of Galena Airport. Calibration information is summarized for each analytical method.

1.8.3.1 Extraction Methods

Extraction methods for liquid and solid matrices are briefly described in this section.

Method SW3005

Acid Digestion of Aqueous Samples for Analyses by ICPES

This method is an acid digestion procedure for the preparation of water samples for metals analysis. The digested samples can be analyzed for total recoverable metals determination by inductively

Table 1.8-1
Analytical Methods to be Used During the Stage 3, RI/FS Activities at Galena AFS

Parameter	Analytical Method	
	Water	Soil
ICP Metals Screen (23 metals less B and Si)	SW3005/SW6010	SW3050/SW6010
Arsenic	SW3020/SW7060	NA
Lead	SW3020/SW7421	NA
Organochlorine Pesticides and PCBs	SW3510/SW8080	SW3540/SW8080
Volatile Organic Compounds	SW5030/SW8260	SW5030/SW8240
Semivolatile Organic Compounds	SW3510/SW8270	SW3540/SW8270
Dioxins and Furans	NA	SW3550/SW8280
Purgeable Petroleum Hydrocarbons	Alaska Method AK101	Alaska Method AK101
Extractable Petroleum Hydrocarbons	Alaska Method AK102	Alaska Method AK102
Soil Moisture Content	NA	ASTM D2216 (modified)

NA = Not Applicable.

Table 1.8-2
Quantitation Limits for Trace Elements (Metals) for GFAA and ICPES

Method	Parameter	Analytes	Practical Quantitation Limits[a]	
			Water (mg/L)	Soil (mg/kg)[b]
SW7060	GFAA Metals	Arsenic	0.004	NA
SW7421	GFAA Metals	Lead	0.005	NA
SW6010	ICP Metals	Aluminum	0.5	50
		Antimony	0.4	40
		Arsenic	NA	60
		Barium	0.02	2
		Beryllium	0.003	0.3
		Cadmium	0.04	4
		Calcium	1	100
		Chromium	0.07	7
		Cobalt	0.07	7
		Copper	0.06	6
		Iron	0.07	7
		Lead	NA	50
		Magnesium	1	100
		Manganese	0.02	2
		Molybdenum	0.08	8
		Nickel	0.15	15
		Potassium	5	100
		Selenium	0.8	80
		Silver	0.07	7
		Sodium	1	100
		Thallium	0.4	40
		Vanadium	0.08	8
		Zinc	0.02	2

[a]The Practical Quantitation Limits are taken from the 1991 IRP Handbook. Specific quantitation limits are highly matrix dependent.
[b]Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

SW = Methods from SW846, Third Edition.
GFAA = Graphite Furnace Atomic Absorption.
NA = Not applicable.

Table 1.8-3
Quantitation Limits for Method SW8080
Organochlorine Pesticides and PCBs

Method	Parameter	Analytes	Practical Quantitation Limits[a, b]	
			Water (µg/L)	Low Soil/ Sediment (mg/kg)
SW8080	Organochlorine Pesticides and PCBs	Alpha-BHC	0.03	0.002
		Beta-BHC	0.06	0.005
		Delta-BHC	0.09	0.0016
		Gamma-BHC (Lindane)	0.04	0.003
		Heptachlor	0.03	0.002
		Aldrin	0.04	0.003
		Heptachlor epoxide	0.83	0.06
		Endosulfan I	0.14	0.009
		Dieldrin	0.20	0.01
		4,4-DDT	0.12	0.008
		Endrin	0.06	0.004
		Endosulfan II	0.04	0.003
		4,4-DDD	0.11	0.007
		Endosulfan sulfate	0.66	0.04
		4,4-DDE	0.04	0.003
		Methoxychlor	1.76	0.1
		Endrin aldehyde	0.23	0.02
		Chlordane	0.14	0.009
		Toxaphene	2.4	0.2
		Aroclor-1016	1.0	1.0
		Aroclor-1221	1.0	1.0
		Aroclor-1232	1.0	1.0
		Aroclor-1242	1.0	1.0
		Aroclor-1248	1.0	1.0
		Aroclor-1254	1.0	1.0
		Aroclor-1260	1.0	1.0

[a]The Practical Quantitation Limits are taken from the 1991 IRP Handbook. Specific quantitation limits are highly matrix dependent.

[b]Sensitivity of the method depends on the level of interference rather than instrumental limitations. Typical waste samples may have higher reporting limits and may require additional cleanup techniques.

Table 1.8-4
Quantitation Limits for Method SW8240
Volatile Organic Compounds

Method	Parameter	Analytes	Practical Quantitation Limits[a] Soil (mg/kg)[b, c]
SW8240	Volatile Organic Compounds	Acetone	0.10
		Benzene	0.005
		Bromodichloromethane	0.005
		Bromoform	0.005
		Bromomethane	0.01
		2-Butanone (MEK)	0.10
		Carbon disulfide	0.005
		Carbon tetrachloride	0.005
		Chlorobenzene	0.005
		Chloroethane	0.01
		2-Chloroethyl vinyl ether	0.01
		Chloroform	0.005
		Chloromethane	0.01
		Dibromochloromethane	0.005
		1,1-Dichloroethane	0.005
		1,2-Dichloroethane	0.005
		1,1-Dichloroethene	0.005
		cis-1,2-Dichloroethene	0.005
		trans-1,2-Dichloroethene	0.005
		1,2-Dichloropropane	0.005
		trans-1,3-Dichloropropene	0.005
		cis-1,3-Dichloropropene	0.005
		Ethylbenzene	0.005
		2-Hexanone	0.05
		Methylene chloride	0.005
		4-Methyl-2-pentanone	0.05
		Styrene	0.005
		Tetrachloroethene	0.005
		1,1,2,2-Tetrachloroethane	0.005
		Toluene	0.005
		1,1,1-Trichloroethane	0.005
		1,1,2-Trichloroethane	0.005
		Trichloroethene	0.005
		Vinyl acetate	0.05
		Vinyl chloride	0.01
		Total xylenes	0.005

[a]The Practical Quantitation Limits are taken from the 1991 IRP Handbook. Specific quantitation limits are highly matrix dependent.

[b]Quantitation limits are for the low level method. Mid- and high-level methods will have corresponding adjustment in limits. These specific limits will be verified by matrix spiking.

[c]Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil sediment, calculated on a dry weight basis as required by the contract, will be higher.

SW = Methods from SW846, Third Edition.

Table 1.8-5
Quantitation Limits for Method SW8260
Volatile Organic Compounds

Method	Parameter	Analytes	Practical Quantitation Limits[a] Water (µg/L)
SW8260	Volatile Organic Compounds	Acetone	20
		Benzene	1
		Bromobenzene	1
		Bromoform	1
		Bromomethane	1
		Bromodichloromethane	1
		2-Butanone (MEK)	2
		Carbon disulfide	20
		Carbon tetrachloride	1
		Chlorobenzene	1
		Chloroethane	1
		2-Chloroethyl vinyl ether	2
		Chloroform	10
		1-Chlorohexane	1
		Chloromethane	1
		Dibromochloromethane	2
		Dibromomethane	1
		1,2-Dichlorobenzene	1
		1,3-Dichlorobenzene	1
		1,4-Dichlorobenzene	1
		1,1-Dichloroethane	1
		1,2-Dichloroethane	1
		1,1-Dichloroethene	1
		cis-1,2-Dichloroethene	1
		trans-1,2-Dichloroethene	1
		1,2-Dichloropropane	1
		trans-1,3-Dichloropropene	1
		cis-1,3-Dichloropropene	1
		Ethylbenzene	1
		2-Hexanone	1
		Methylene chloride	5
		Methyl isobutyl ketone	2
		Styrene	20
		1,1,1,2-Tetrachloroethane	1
		Tetrachloroethene	1
		1,1,2,2-Tetrachloroethane	1
		Toluene	1
		Trichlorofluoromethane	1
		1,1,1-Trichloroethane	2
		1,1,2-Trichloroethane	1
		1,2,3-Trichloropropane	1
		Trichloroethene	1
		Vinyl acetate	1
		Vinyl chloride	2
		m-Xylene[b]	1
		o-Xylene	1
		p-Xylene[b]	1

[a]The Practical Quantitation Limits are taken from the 1991 IRP Handbook. Specific quantitation limits are highly matrix dependent.

[b]m&p Xylene co-elute.

SW = Methods from SW846, Third Edition.

Table 1.8-6 (Continued)

Method	Parameter	Analytes	Practical Quantitation Limits[a]	
			Water (µg/L)	Soil (mg/kg)[b]
Base/Neutral Extractables (Continued)				
SW8270		Indeno(1,2,3-cd)pyrene	10	0.7
		Isophorone	10	0.7
		2-Methylnaphthalene	10	0.7
		Naphthalene	10	0.7
		2-Nitroaniline	50	3.3
		3-Nitroaniline	50	3.3
		4-Nitroaniline	50	3.3
		Nitrobenzene	10	0.7
		n-Nitrosodiphenylamine[c]	10	0.7
		n-Nitrosodipropylamine	10	0.7
		Phenanthrene	10	0.7
		Pyrene	10	0.7
		1,2,4-Trichlorobenzene	10	0.7
Acid Extractables				
SW8270		Benzoic acid	50	1.7
		4-Chloro-3-methylphenol	20	1.3
		2-Chlorophenol	10	0.33
		2,4-Dichlorophenol	10	0.33
		2,4-Dimethylphenol	10	0.33
		4,6-Dinitro-2-methylphenol	50	3.3
		2,4-Dinitrophenol	50	3.3
		2-Methylphenol	10	0.33
		4-Methylphenol	10	0.33
		2-Nitrophenol	10	0.33
		4-Nitrophenol	50	1.6
		Pentachlorophenol	50	3.3
		Phenol	10	0.33
		2,4,5-Trichlorophenol	50	3.3
		2,4,6-Trichlorophenol	10	0.33

[a]The Practical Quantitation Limits are taken from the 1991 IRP Handbook. Specific quantitation limits are highly matrix dependent.

[b]Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

[c]n-Nitrosodiphenylamine decomposes to diphenylamine. Laboratory quantitates as diphenylamine.

SW = Methods from SW846, Third Edition.

Table 1.8-7
Quantitation Limits for Method SW8280
Polychlorinated Dioxins and Furans

Method	Parameter	Analytes	Practical Quantitation Limits[a] (mg/kg)[b,c]
SW8280	Polychlorinated Dioxins/Furans	2,3,7,8-TCDD	0.00017
		TCDD	0.00017
		TCDF	0.00017
		PeCDD	0.00018
		PeCDF	0.00017
		HxCDD	0.00018
		HxCDF	0.00017
		HpCDD	0.00024
		HpCDF	0.00017
		OCDD	0.00032
		OCDF	0.00023

[a]The Practical Quantitation Limits are taken from the 1991 IRP Handbook. Specific quantitation limits are highly matrix dependent.

[b]Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

SW = Methods from SW846, Third Edition.

Table 1.8-8
Quantitation Limits for Field Soil Test Kits

Parameter	Analytes	Lower Limit of Detection[a] Soil/Sediment (mg/kg)
PCB Test Kit	PCBs	3.3 for standard protocol 0.5 for high sensitivity protocol
DDT Test Kit	4,4'-DDT	0.04
	4,4'-DDD	0.01
	4,4'-DDE	0.18
	1,4'-DDT	4.0
	1,4'-DDD	0.4
	1,4'-DDE	3.0

[a]Manufacturer recommended values for Millipore EnviroGard™ DDT Soil Test Kit and PCB Soil Test Kit

coupled plasma emission spectroscopy (ICPES). Samples will analyzed for the following metals:

Aluminum	Copper	Silver
Antimony	Iron	Sodium
Barium	Magnesium	Thallium
Beryllium	Manganese	Vanadium
Cadmium	Molybdenum	Zinc
Calcium	Nickel	
Chromium	Potassium	
Cobalt	Selenium	

For analysis of total recoverable metals, the entire sample is acidified at collection time with nitric (HNO_3) acid to a pH < 2. At the time of analysis, a 50-mL aliquot of the sample is heated with 1 mL of concentrated nitric acid and 5 mL of 1:1 hydrochloric acid and reduced to a specific volume. The sample must not be boiled because antimony is volatile and easily lost. The digestate is then filtered and adjusted to a final volume of 50 mL with reagent water.

Modified Method SW3020
Acid Digestion of Aqueous Samples for Arsenic and Lead Analyses by Graphite Furnace Atomic Absorption Spectroscopy

Water samples will be digested according to the method SW3020 which has been modified by adding hydrogen peroxide to facilitate digestion efficiency and elimination of organic interferences. In the modified method SW3020, a mixture of the sample, nitric acid, and hydrogen peroxide is heated in a Griffin beaker. This step is repeated with an additional portion of nitric acid and is refluxed until the digestate is light in color or until its color has stabilized. After the sample digestion is complete, the digestate is brought up to the original volume with deionized water. Matrix modifiers are added prior to analysis as appropriate for the element of interest.

Method SW3050
Acid Digestion for Solids, Sediments, and Sludges for Metals Determinations

Method SW3050 is applicable to the preparation of sediment, sludge, and soil samples for metals analysis by FLAA or GFAA or ICPES.

A 1 g (wet weight) sample is treated and digested in nitric acid and hydrogen peroxide. The digestate is then refluxed with nitric or hydrochloric acid, depending on the type of analysis to be performed. When using hydrochloric acid as the final refluxing acid, care is taken not to boil the solution because antimony is volatile and easily lost. A separate sample is dried for a total solids and/or percent moisture determination.

Some sludge samples can contain diverse matrix types, which may present specific analytical problems. Spiked samples and any relevant standard reference material should be processed to aid in determining whether Method SW3050 is applicable to a given waste.

SW3500 Series Methods
Organic Extraction and Sample Preparation

The SW3500 series methods are used to quantitatively extract nonvolatile and semivolatile organic compounds from various sample matrices. Prior to analysis, a sample of a known volume or weight is solvent extracted, then dried and concentrated in a Kuderna-Danish apparatus.

Method SW3510
Separatory Funnel Liquid-Liquid Extraction

Method SW3510 is designed to quantitatively extract nonvolatile and semi-volatile organic compounds from liquid samples using standard separatory funnel techniques. The sample and extracting solvent must be immiscible in order to yield recovery of target compounds. Subsequent cleanup and detection methods are described in the organic analytical method that will be used to analyze the extract.

Samples are adjusted to a specified extraction pH and extracted with the appropriate solvent for the analytical method. Methylene chloride should be employed when a solvent is not specified.

Method SW3520
Continuous Liquid-Liquid Separatory Extraction

point. Determinations will be made according to indicator color change (phenolphthalein, metacresol purple, or bromcresol green). Concentrations of the hydroxide, carbonate, and bicarbonate alkalinity are determined by the relationship of phenolphthalein alkalinity (at pH 8.3) to the total alkalinity (at pH 4.5).

Method SW9050
Specific Conductance

Sample conductance is measured using Method SW9050. Standard field meters are used on site and the electrode is rinsed with sample prior to measuring conductance; temperature is also reported. The meters are standardized daily using KCl solutions of known conductance with an allowance of ±5% of true value. Method detection limits are presented in Table 1.8-2.

Methods SW9040/SW9045
pH

Field pH measurements will be taken for water samples: Measurements are determined electrometrically using either a glass electrode in combination with a reference potential, or a combination electrode. The meters are calibrated daily using a minimum of two buffer solutions. The calibration readings must be within 0.05 units of the known buffer pH.

EPA Method 170.1
Temperature

On-site water temperature is measured using EPA Method 170.1. A factory-calibrated standard mercury thermometer is rinsed twice with sample prior to recording the temperature.

GAC Method for TRPH
TRPH Screening Method

Soil samples will be analyzed for TRPH in the field laboratory using a field extraction procedure followed by analysis using the GAC TRPH meter. Samples are prepared by mixing a measured amount of soil or water with Freon-113™. The sample is shaken, allowed to settle, and the freon layer is then filtered through a particle filter and a silica gel cartridge. The resultant extract is then placed in a 10 mm cuvette and analyzed using the GAC TRPH meter. The concentration of TRPH (mg/kg) in the sample is then calculated from the digital display (mg/L) times the volume of solvent (L) used and divided by the sample weight or volume (kg).

Graphite Furnace Atomic Absorption Metals Analyses, SW7060 for Arsenic and SW7421 for Lead

Graphite furnace AA spectrometry will be used to measure concentrations of arsenic and lead in water samples. The water samples are digested using SW3020 (modified). Discrete aliquots of sample extract are deposited in a graphite tube furnace in microliter amounts. The graphite tube is resistively heated by an electrical current. The sample solution is dried and charred to remove sample matrix components, and then atomized at temperatures sufficient to vaporize the element of interest. Matrix modification is used to eliminate interference effects, and may also enhance the vaporization efficiency. This method usually has a linear analysis range at the µg/L or sub-µg/L level. Quantitation limits for this arsenic and lead are presented in Table 1.8-2.

Calibration—The calibration procedures for the graphite furnace AA systems are described in the respective methods in SW846, Third Edition. A multi-point calibration curve is generated daily for each element using a calibration blank and at least three upscale standards. The correlation coefficient for the linear regression equation must be ≥ 0.995 to be acceptable. Calibration will be verified every 10 samples by analyzing a continuing calibration verification sample and calibration blank. Agreement within ±20 percent of the expected value is required; otherwise, a new calibration curve must be generated.

Method SW6010
Metals by ICPES for Soil

Samples are analyzed for metals using SW6010 for soils. Analysis for most metals requires digestion of the sample with acid. This digestion is performed as SW846 Method 3050 for soil. Following digestion, the trace elements are simultaneously or sequentially determined using ICPES. The elements and corresponding reporting limits for this method are listed in Table 1.8-2.

Calibration—Detailed calibration procedures for ICPES systems are described in SW846, Third Edition. A response factor is calculated daily for each metal based on three exposures of a calibration standard and calibration blank. The RF is calculated and stored in the ICPES computer. Following calibration, a mid-level calibration check sample is analyzed; agreement between the measured value and the expected value must be within 5% for analyses to proceed. Calibration is verified by analyzing a QC check standard (prepared independently of calibration standards) every 10 samples; agreement within ±10% of the expected value is required for all metals analyzed by ICPES.

Method SW8080
Organochlorine Pesticides and PCBs

Organochlorine pesticides and polychlorinated biphenyls (PCBs) in water and soil samples are analyzed using Method SW8080. This analytical method involves extraction of the sample with methylene chloride, followed by exchange to hexane and concentration of the extract. The pesticides and PCBs are separated and quantified by gas chromatography using electron capture detection. Both neat and diluted liquids may be analyzed by direct injection on to the chromatographic column. Quantitation limits for this method are presented in Table 1.8-3.

Calibration—The external standard quantitation discussed in the method is used to quantitate all pesticides/PCBs. The retention time window is calculated for each pesticide/PCB after adjusting the GC operating conditions for the routine retention times. The GC/ECD is initially calibrated at a minimum of five concentrations. The average calibration factor is acceptable if the relative standard deviation (RSD) for the calibration factors at each level does not exceed 20% or linear regression may be used if the correlation coefficient is 0.995 or greater. Concentration of the components in a standard varies depending on the response of the compounds in the analytical system. Breakdown of 4,4'-DDT and endrin is also monitored. Breakdown may not exceed 20 percent. A daily single-point calibration check must agree within ±15% of the multipoint response or the instrument is recalibrated.

Method SW8240
Volatile Organics

Volatile, or purgeable, organics in soil samples are analyzed using Method SW8240. This method uses a purge-and-trap GC/MS technique. An inert gas is bubbled through a soil-water slurry for soil samples to transfer the purgeable organic compounds from the liquid to vapor phase. Soil samples with higher contaminant levels are extracted using methanol before purging. The vapor is then swept through a sorbent trap where the purgeable organics are trapped. The trap is backflushed and heated to desorb the purgeable organics onto a gas chromatographic column where they are separated and then detected with a mass spectrometer. The species detected and quantitation limits for this method are listed in Table 1.8-4.

Calibration—The mass spectrometer is tuned daily to give an acceptable spectrum for bromofluorobenzene (BFB). Relative ion abundance criteria for BFB are given in SW846. System performance is verified initially and after every 12 hours to ensure a minimum average response factor of 0.3 (0.25 for bromoform) for the system performance check compounds (SPCCs). A five-point calibration is used for generating response factors. The RSD must be less than 30% for the five response factors calculated for each of the calibration check compounds (CCCs).

Method SW8260
Volatile Organics

Volatile, or purgeable, organics in water samples are analyzed using Method SW8260. This method uses a purge-and-trap GC/MS technique. An inert gas is bubbled through the liquid samples to transfer the purgeable organic compounds from the liquid to vapor phase. The vapor is then swept through a sorbent trap where the purgeable organics are trapped. The trap is backflushed and heated to desorb the purgeable organics onto a gas chromatographic column where they are separated and then detected with a mass spectrometer. The species detected and quantitation limits for this method are listed in Table 1.8-5.

Calibration—The mass spectrometer is tuned daily to give an acceptable spectrum for bromofluoro-

benzene (BFB). Relative ion abundance criteria for BFB are given in SW846. System performance is verified initially and after every 12 hours to ensure a minimum average response factor of 0.3 (0.25 for bromoform) for the SPCCs. A five-point calibration, used for generating response factors, is performed initially using 3.33, 6.67, 16.7, 33.3 and 66.7 μg/L standards. The RSD must be less than 30% for the five response factors calculated for each of the CCCs.

Method SW8270
Semivolatile Organics Analysis

Semivolatile organics, also known as base/neutral and acid extractables (BNA), in water and soil samples are analyzed using Method SW8270. These techniques quantitatively determine the concentration of a number of semivolatile organic compounds. Organic compounds are extracted from the sample with methylene chloride at pH greater than 12 to obtain base/neutral extractables. Acid extractable compounds are obtained from the sample by extraction with methylene chloride at pH 2 or less. Both base/neutral and acid extracts are then concentrated by removal of the methylene chloride through evaporation. Compounds of interest are separated and quantified using a GC/MS. The compounds that can be detected using Method SW8270 and the quantitation limits are listed in Table 1.8-6.

Calibration—The mass spectrometer is tuned daily to give an acceptable spectrum for decafluorotriphenyl phosphene (DFTPP). Decafluorotriphenyl phosphene ion abundance criteria are given in SW846. System performance is verified initially and after every 12 hours to ensure a minimum average response factor of 0.050 for the SPCCs. A five-point calibration, used for generating response factors. The variability for specific ion response factors for the SW8270 calibration check compounds must be less than 30% RSD over the range calibrated. A continuing (every 12 hours) calibration check is performed, following the system performance check, using the CCCs listed above. A single concentration of each CCC is analyzed and a response factor calculated. The single-point response factor for each CCC must be within 30% of the average five-point response factor; otherwise, a new five-point calibration must be generated.

Method SW8280
Polychlorinated Dioxins and Furans (PCDD/PCDF)

Soil samples are analyzed for polychlorinated dioxins and furans using SW-846 Method 8280. Dioxins and furans are extracted from soil with a combination of hexane and methanol using SW-846 Method 3550. Extracts are cleaned by passing the solvent through an H_2HO_4 shake, alumina, silica gel, and carbon columns. Cleaned extracts are concentrated then injected onto a fused silica capillary column in a GC/MS. Compounds detected and quantitation limits are given in Table 1.8-7.

Calibration—Response factors for each PCDD/PCDF isomer class are determined initially using a five-point calibration, which is performed in triplicate. Average response factors calculated from the triplicate analyses must have a RSD less than 15% for each isomer class. A TCDD chromatographic test mixture is analyzed daily to verify that there is at least 25% valley resolution between 2,3,7,8-TCDD and 1,2,3,4-TCDD. A PCDD/PCDF standard is analyzed daily to verify that the daily response factors are within 30% of the multi-point calibration and that the isotope ratios for each isomer class are within 15% of their theoretical values.

Alaska Method AK102.0
Diesel Range Organics (DRO)

The Alaska Method AK102.0 for diesel range organics (DRO) is designed to measure the concentration of diesel range organics in water and soil. This corresponds to an n-alkane range from the beginning of C_{10} to the beginning of C_{25} and a boiling point range 170°C to 400°C. Samples are spiked with a surrogate and extracted with methylene chloride. The extract is dried and concentrated to a volume of 1.0 mL. An aliquot of the extract is analyzed by injection onto the capillary column of a GC equipped with a flame ionization detector (FID). Quantitation is performed by comparing the total chromatographic area between the peak start of C_{10} and the peak start of C_{24}, includ-

ing resolved and unresolved components, to the response of the diesel calibration standard.

Calibration—A minimum of three-point (five-point is recommended) calibration of the GC is performed using the diesel calibration standard (DCS). The preparation of the DCS is detailed in section 7.4.3 of the method. The recommended surrogate control standard is ortho-terphenyl at a 20 μg/mL working concentration in acetone. A commercial diesel #2 (other than the diesel used to prepare the DCS) at a working concentration of 5000 μg/mL is used for the laboratory control sample.

Alaska Method AK101.0
Gasoline Range Organics (GRO)

The Alaska Method AK101.0 for gasoline range organics (GRO) is designed to measure the concentration of gasoline range organics in water and soil. This corresponds to an alkane peak range from the peak start of C_6 to the peak start of C_{10} and a boiling range between approximately 60°C and 170°C. Water samples are analyzed directly by purge-and-trap gas chromatography (FID or FID/PID). Soil samples are extracted into methanol and a portion of the methanol extract is analyzed by purge-and-trap GC. Quantitation is performed by comparing the total chromatographic area between and including C_6 (hexane) and C_9 (nonane), through the peak start time of C_{10}, including resolved and unresolved components, based on FID response to a blended commercial gasoline standard.

Calibration—A minimum of a three-point (five-point is recommended) calibration of the GC is performed using the gasoline calibration standard (GCS). The GCS is an equal-weight mixture of unleaded, leaded and premium commercial gasolines mixed and diluted to appropriate concentrations. Either bromofluorobenzene or trifluorotoluene, or a mixture of both are used as the surrogate control sample. A reagent blank or method blank sample spiked with a commercial gasoline other than the ones blended to prepare the GCS is used as the laboratory control sample.

1.9 Data Reduction, Validation, and Reporting

The data reduction, validation, and reporting procedures described in this section will ensure that complete documentation is maintained, transcription and data reduction errors are minimized, the quality of the data is reviewed and documented, and the reported results are properly qualified.

1.9.1 Data Management

The primary data management activities for the Galena Airport project will include:

- Data transfer from field and laboratory activities to a project filing system;
- Data management to ensure that data are stored and output in a manner that continues the chain of custody;
- Review requirements to ensure that plans for data collection were fulfilled;
- Analytical data validation of data used for site interpretation activities;
- Analytical and field data evaluation resulting in a report of guidance to be followed for using project data in site interpretation, risk assessment, etc.; and
- Reporting functions which include, but are not limited to, outputting data for report tables, statistical analysis, site interpretation, risk assessment, and electronic transfer to IRPIMS (Installation Restoration Program Information Management System).

A computerized project database will be used for data management on the Galena Airport project. The proposed database will be implemented in a relational data management software and will be based upon project databases used for historical Air Force projects.

1.9.2 Data Reduction

Data reduction activities described in this section are applicable to Radian Analytical Services.

The laboratory analyst is responsible for the reduction of raw data generated at the laboratory bench. The data interpretation that is required to calculate sample concentrations follows the methodology described in the specific analytical standard operating procedure (SOP). After all analyses have been completed and reported, the laboratory manager or designee reviews the raw data and verifies that the analyses were properly performed and reported. The laboratory manager may then transfer the raw data to the document control area, where the raw data are filed for a subsequent QC review by the document control group. Raw data, together with all supporting documentation, are stored in confidential files by document control.

After all analyses for a report are complete, the data are entered into the laboratory reporting system and a preliminary report is generated for review by the laboratory managers. This review is followed by a quality check carried out by the document control group to verify that the QC meets the specifications of the method.

Identification of outliers is also a part of the data review. An outlier is an unusually large (or small) value in a set of observations. There are many possible reasons for outliers including:

- Faulty instruments or component parts;
- Inaccurate reading of a record, etc;
- Errors in transcribing data; and
- Calculation errors.

Sometimes analysts or operators can identify outliers by noting the above types of occurrences when they record observations. In these instances, the errors are corrected, or if correction is not possible, the suspect observations may be removed from the data before calculations are performed. If no such information exists, the Dixon Criteria are used to test suspected outliers at the 5% significance level for data sets containing three or more points (Dixon, 1953). Outliers identified by this method may be removed from the data before further processing.

1.9.3 Data Quality Assessment

Data validation activities for the Galena Airport project will approximate activities followed for an EPA Level III project (EPA, 1987). The following summarizes the expected level of effort for a Level III project. Data validation will be performed by laboratory staff at the laboratories standard level of effort. This standard level of effort includes:

- Peer review of natural matrix and QC measurement data at the analyst level;
- Preliminary report review by laboratory managers; and
- A quality check performed by the laboratory's document control group.

The QA coordinator, or a designee, will validate data by reviewing field and laboratory documentation and measurement data for acceptable sample collection and analysis procedures, consistency with expected results or other results, adherence to prescribed QA procedures, and agreement with the acceptance criteria described in section 1.0, including:

- Review approximately 10% of all analytical batches for conformance with internal QC requirements stipulated in section 1.10 of this SAP Addendum.
- Review all results for holding time compliance.
- Laboratory control charts may be reviewed for compliance with established criteria. The control chart review may be conducted periodically during site visits or audits. Control charts for field duplicate results can be developed to document systematic precision for a method.

Validated data will be reported in the Informal Technical Information Report (ITIR) and used for

data analysis activities such as site interpretation and risk assessment.

1.9.4 Data Evaluation and Reporting

The project QA coordinator, or other QA staff, will review and summarize all QC sample results to evaluate the sampling and analytical performance. Reagent and field blank results will be evaluated to identify any systematic contamination; spike and duplicate results will be compared to the QA objectives presented in section 1.0 and the results used to calculate precision and accuracy for the data set. This process will identify analytical methods and compounds for which the QA objectives are not satisfied, and corresponding sample data will be qualified with a "flag" indicating the problem. Samples collected on the same day, or analyzed in the same run or batch, or individual samples may be flagged, depending on the type of problem that has been identified. Re-analysis or resampling may be recommended as a corrective action if data are determined to be unacceptable for the intended application. Corrective actions and data assessment procedures are described in sections 1.13 and 1.14. The specific statistical procedures and qualifier codes used in the evaluation process are described in detail in section 1.13.

Data reporting for this project will consist of IRP reporting. General reporting practices for measurement data will include:

- Heading information identifying the sample ID and the analytical method, as provided on laboratory reports;
- Unique sample identification number or code;
- Consistent units of measure;
- Consistent number of significant figures; and
- Comparison with regulatory threshold values.

QC results will be reported by sample matrix and analytical method in tabular form. The influence of these QC results on the measurement data will be delineated. For example, matrix spike interference will influence specific samples or matrices, while laboratory blank contamination will influence all samples extracted or analyzed on a specific day or during a specific analytical run.

When a large number of QC analyses of one type exists, a summary table will be constructed. The summary tables will typically report mean or pooled statistics to describe the overall performance of the method. For example, the summary table of duplicate sample results might report the average RPD for all duplicates measured for the compound, and indicate the number of individual RPDs that did not meet the acceptance criteria. This type of summary can be an indication of the overall QC results. However, these applications will often have to be developed or modified from existing programs for individual investigations. A summary assessment of the data presented in these tables will be prepared for each phase of sampling, or specific RI/FS task, as appropriate.

Finally, custom table formats will be used as an aid to interpretation of the investigative data. The particular format will depend on how the QC results are expected to influence the investigative data and will be developed by data management staff through discussion with the users. For example, QC results may be grouped with analytical batches, field collection batches, or summarized for the entire project.

1.10 Internal Quality Control Checks for Field and Laboratory Operations

Internal QC is achieved by collecting and/or analyzing a series of duplicate, replicate, blank, spike, and spike duplicate samples to ensure that the analytical results are within QC limits specified for the program. Laboratory QC samples are documented at the bench and reported with the analytical results. The QC sample results are used to quantify precision and accuracy and identify any problems or limitations in the associated sample results. The specific number of normal samples, field duplicates, equipment blanks, trip blanks, ambient blanks and MS/MSD samples for analytical method are summarized in Table 1.10-1.

1.10.1 Field Quality Control

Field QC samples will be documented in field logbooks and submitted "blind" to the laboratory, so that the laboratory cannot distinguish between natural and QC samples during analysis. These components of the sampling program will ensure that data of known quality are produced throughout the sampling and analysis component of all field programs.

1.10.1.1 Field Duplicate Samples

A field duplicate sample is a second sample collected at the same location as the original sample. Duplicate sample results are used to assess precision, including variability associated with both the laboratory analysis and the sample collection process. Duplicate samples will be collected simultaneously or in immediate succession, using identical recovery techniques, and treated in an identical manner during storage, transportation, and analysis. Duplicate water samples will be collected at a frequency of 10 percent. Field duplicate soil samples will not be taken for any analysis, per the directive of the AFCEE.

1.10.1.2 Ambient Blank

Ambient blanks are samples of reagent grade water that are poured in the field and processed using the same sampling and handling procedures as other samples. Ambient blanks are used to assess the potential introduction of contaminants to the samples during sample collection and analysis, and are prepared only for VOC samples. Ambient blanks (estimated five samples) will be taken at any groundwater sampling locations which are sampled downwind of unavoidable VOC sources, such as active roadways or runways.

1.10.1.3 Equipment Blanks

Equipment blanks consist of a sample of Type II reagent water or organic-free water (for volatile organic analyses) poured into the groundwater or soil sampling device, collected in the sample bottle, and transported to the laboratory for analysis. Equipment blanks for Galena groundwater sampling will be taken only if non-dedicated sampling equipment is used. Equipment blanks for soil samples will be collected at a minimum rate of 10%.

1.10.1.4 Trip Blanks

A trip blank is a sample of organic-free water that is placed in the sample bottle in an uncontaminated area in the laboratory prior to going in the field. Trip blanks are prepared only for VOC samples and are subjected to the same handling as other samples. Trip blanks serve to identify contamination from sample containers or transportation and storage procedures. One trip blank will accompany each shipment or cooler (estimated that of ten trip blanks) of Galena water samples sent to the laboratory for the analysis of VOCs and AK101.0 GRO samples. The trip blank for soil samples will be the same as for water samples. Organic-free water will be prepared with reagent grade water that has been filtered, deionized, and boiled to volatilize organic compounds. This water is then continuously purged with nitrogen to prevent re-entry of volatile organic compounds.

1.10.2 Laboratory Quality Control

Laboratory QC is necessary to control the analytical process, to assess the accuracy and precision of analytical results, and to identify assignable causes for atypical analytical results. The QC checks in the laboratory protocol are specific to the analytical method and generally include the use of the following QC samples as appropriate for the method.

1.10.2.1 Calibration Standards

Initial calibration is performed as required for each analytical method, usually using a range of calibration standards with the low standard near the detection limit for the compound. These standards are used to determine the linear dynamic range for the initial instrument calibration. EPA, NIST, CRADA (Cooperative Research and Development Agreement) or other approved standards will be used when possible. Calibration is discussed in more detail in sections 1.7 and 1.8.

1.10.2.2 Laboratory Control Samples

Laboratory control samples (LCS) are check solutions containing certain analytes of interest (including all target analytes except for 8080 analyses) at a specified concentration, usually in the mid-calibration range. These samples are used to demonstrate

Table 1.10-1
Field Collected Laboratory and QC Samples for Kalakaket RRS and Galena Airport

Parameter	Method	Normal Samples	Field Duplicates	Equipment Blanks	Trip Blanks	Ambient Blanks	MS/MSD	Total Samples
Kalakaket Creek RRS Soil Samples								
Metals by ICPES	SW6010	21	0	3	0	0	4	28
Petroleum Hydrocarbons—purgeable	AK101	22	0	3	0	0	4	29
Volatile Organics	SW5030/SW8240	17	0	2	0	0	2	21
Petroleum Hydrocarbons—extractable	AK102	22	0	3	0	0	4	29
Semivolatile Organics	SW3540/SW8270	14	0	2	0	0	2	18
Pesticides and PCBs	SW3540/SW8080	17	0	2	0	0	2	21
Galena Airport Water Samples								
Metals by ICPES	SW6010	6	1	1[a]	0	0	2	10
As by GFAAs	SW7060	6	1	1[a]	0	0	2	10
Pb by GFAAs	SW7421	6	1	1[a]	0	0	2	10
Petroleum Hydrocarbons—purgeable	AK101	41	5	1[a]	10[b]	5	6	70
Volatile Organics	SW5030/SW8260	41	5	1[a]	10[b]	5	6	70
Petroleum Hydrocarbons—extractable	AK102	41	5	1[a]	0	0	6	55
Semivolatile Organics	SW3510/SW8270	35	4	1[a]	0	0	6	55
Pesticides and PCBs	SW3510/SW8080	41	5	1[a]	0	0	6	55
Galena Airport Soil Samples								
Metals by ICPES	SW6010	4	0	1	0	0	2	7
Petroleum Hydrocarbons—purgeable	AK101	4	0	1	0	0	2	7
Volatile Organics	SW5030/SW8240	4	0	1	0	0	2	7
Petroleum Hydrocarbons—extractable	AK102	4	0	1	0	0	2	7
Semivolatile Organics	SW3540/SW8270	4	0	1	0	0	2	7
Pesticides and PCBs	SW3540/SW8080	34	0	4	0	0	4	42
Dioxins and Furans	SW3510/SW8280	8	0	1	0	0	0	9

[a] A groundwater equipment blank will be collected only if non-dedicated equipment is used.
[b] Trip blank number includes those needed for soil samples.

that the instrument and method is operating within acceptable accuracy limits. LCSs are required for all analytical methods performed in the laboratory, and their preparation and the required frequency of analysis is described in each analytical SOP. For the Galena Airport project, acceptance criteria pertain to liquid LCSs. Liquid LCSs will determine whether or not the analytical system is capable and in control.

1.10.2.3 Method Blanks

A reagent or method blank is a sample composed of all the reagents (in the same quantities) used in preparing a sample for analysis. It is carried through the same sample preparation (digestion, extraction) procedure as a sample. As stated in the SW846 Method 8000 procedures, reagent blanks are used to ensure that interferences from the analytical system, reagents, and glassware are under control. The required frequency for analyzing reagent blanks is specified in the analytical SOP for each method and generally consists of one per day for each method/instrument and/or one per extraction batch.

1.10.2.4 Matrix Spike/Matrix Spike Duplicates

A matrix spike is a solution of target analytes at known concentrations that is spiked into a field sample before sample preparation and analysis. Two aliquots of the sample are spiked for the duplicate analysis. The results of the analysis of the duplicate spiked samples are used to measure the percent recovery of each spiked compound and to compare the recovery between samples, which provides estimates of the accuracy and precision of the method. The calculations for accuracy and precision are outlined in section 1.13. The frequency for matrix spike analysis is 5% of samples analyzed for each method where spikes are performed. The solution of target analytes in matrix spikes for metals includes all target analytes. The solution of target analytes in matrix spikes for organic analyses is based on Method 3500 and does not include all target analytes, but is rather a representative subset.

1.10.2.5 Surrogate Spikes

Surrogate spikes are a group of compounds that are not otherwise found in nature but behave similarly to the target analytes that have been selected for each organic analytical method. A solution of known concentration is prepared and spiked into each sample prior to sample preparation and analysis. The recovery of the surrogate spike compounds is reported for each sample, and the results are compared to the recovery objectives established for the method. Surrogate compounds are recommended in each analytical method for organic constituents. Where feasible, recommended surrogates will be used.

1.10.2.6 Laboratory Duplicates (Duplicate Analyses)

Laboratory duplicates are repeated but independent determinations of the same sample, by the same analyst, at essentially the same time and under the same conditions. The sample is split in the laboratory and each fraction is carried through all stages of sample preparation and analysis. Duplicate analyses measure the precision of each analytical method. The method of calculation for precision is outlined in section 1.13. Laboratory duplicate analyses are performed for 10% of samples analyzed, or at least one per day, for analytical methods that do not require matrix spike-matrix spike duplicates.

1.10.3 Control Limits

Control limits and acceptance criteria for QC samples are presented by method in Tables 1.10-2 through 1.10-8.

1.11 Performance and System Audits

A performance or system audit is not planned for this current sampling effort. Audits will not be performed because these activities are not scoped in the SOW.

1.12 Preventative Maintenance

The primary objective of a preventative maintenance program is to promote the timely and effective completion of a measurement effort. The preventative maintenance program is designed to minimize the downtime of crucial sampling and/or analytical equipment from component failure. Implementation of a preventative maintenance program requires the establishment of:

Table 1.10-2
Quality Control Acceptance Criteria Indicator Parameters

Parameter	Aqueous Matrix Spike		Solid Matrix Spike		LCS Spike
	Accuracy[a] (% Recovery)	Precision (RPD)	Accuracy[a] (% Recovery)	Precision (RPD)	Accuracy[a] (% Recovery)
Soil Moisture	NA	NA	NA	30[b]	NA
AK102.0 Diesel Range Organics DCS	60 - 120	≤20	60 - 120	≤20	75-125
AK101.0 Gasoline Range Organics GCS	60 - 120	≤20	60 - 120	≤20	75-125
Surrogate Diesel Range Organics Ortho-terphenyl (OTP)	60 - 120	NA	60 - 120	NA	
Gasoline Range Organics Trifluorotoluene or Bromofluorobenzene	60 - 120	NA	60 - 120	NA	

[a] These are established limits. *MS/MSD results are used for post-analysis, project review; LCS results are used for control of the analytical system.*

[b] Based on analytical duplicates instead of matrix spiked duplicates.

NA = Not applicable.
RPD = Relative percent difference.
LCS = Laboratory control sample.
DCS = Diesel Calibration Standard
GCS = Gasoline Calibration Standard

Table 1.10-3
Quality Control Acceptance Criteria for Metals

Parameter	Aqueous Matrix Spike[a]		Solid Matrix Spike[a]		LCS Spike[b]
	Accuracy (% Recovery)	Precision (RPD)	Accuracy (% Recovery)	Precision (RPD)	Accuracy (% Recovery)
GFAA Arsenic SW7060	75-125	20	NA	NA	75-125
GFAA Lead SW7421	75-125	20	NA	NA	75-125
ICP Metals SW6010	75-125	20	75-125	35	80-120

[a] Reference guide for post-analysis project review.

[b] Used for control of analytical system.

RPD = Relative percent difference.
LCS = Laboratory control sample.

NA = Not applicable.

Table 1.10-4
Quality Control Acceptance Criteria for Organochlorine Pesticides and PCBs by Method SW8080[a]

Parameter	Aqueous Matrix Spike		Solid Matrix Spike		LCS Spike
	Accuracy[b] (% Recovery)	Precision (RPD)	Accuracy[b] (% Recovery)	Precision (RPD)	Accuracy[b] (% Recovery)
Aldrin	42 - 122	≤55	42 - 122	≤55	42 - 122
alpha-BHC					37 - 134
Chlordane					NA
alpha-Chlordane					NS
gamma-Chlordane					NS
4,4'-DDD					NS
4,4'-DDE					NS
4,4'-DDT					25 - 160
delta-BHC	25 - 160	≤85	25 - 160	≤85	19 - 140
Dieldrin					36 - 146
Endosulfan I	36 - 146	≤44	36 - 146	≤44	NS
Endosulfan II					D - 202
Endosulfan sulfate					NS
Endrin					30 - 147
Endrin aldehyde	30 - 147	≤67	30 - 147	≤67	NS
Endrin ketone					NA
gamma-BHC (Lindane)					32 - 127
Heptachlor	32 - 127	≤63	32 - 127	≤63	34 - 120
Heptachlor epoxide	34 - 120	≤43	34 - 120	≤43	37 - 142
Methoxychlor					NS
Mirex					NS
PCB-1016					50 - 120
PCB-1221	50-120	≤41			NS
PCB-1232					NS
PCB-1242					NS
PCB-1248					NS
PCB-1254					NS
PCB-1260					8 - 127
Toxaphene	8-127	≤102			NS
Surrogates					
Dibutylchlorendate	20 - 150	NA	20 - 150	NA	
TCMX	20 - 150	NA	20 - 150	NA	

[a]All limits are based on a certain spike concentration. If this concentration changes, the limits also change. See Tables 3 and 4 in SW8080.

[b]Method specified limits from Chapter One, SW-846 and tables in method. Also, the LCS will be spiked at a minimum with the matrix spike analytes. *Ms/MSD results are used for post-analysis, project review; LCS results are used for control of the analytical system.*

RPD = Relative percent difference.
NS = Not spiked.
LCS = Laboratory control sample.

Table 1.10-5
Quality Control Acceptance Criteria for Volatile Organic Compounds by Method SW8240[a]

Parameter	Solid Matrix Spike		LCS Spike
	Accuracy[b] (% Recovery)	Precision (RPD)	Accuracy[b] (% Recovery)
Acetone			NS
Benzene	37 - 151	≤60	37 - 151
Bromodichloromethane			35 - 155
Bromoform			45 - 169
Bromomethane			D - 242
2-Butanone (MEK)			NS
Carbon disulfide			NS
Carbon tetrachloride			70 - 140
Chlorobenzene	37 - 160	≤63	37 - 160
Chloroethane			NS
2-Chloroethyl vinyl ether			NS
Chloroform			51 - 138
Chloromethane			D - 273
Dibromochloromethane			53 - 149
1,1-Dichloroethane			59 - 155
1,2-Dichloroethane			49 - 155
1,1-Dichloroethene	D - 234	≤133	D - 234
trans-1,2-Dichloroethene			54 - 156
1,2-Dichloropropane			D - 210
trans-1,3-Dichloropropene			17 - 183
cis-1,3-Dichloropropene			D - 227
Ethylbenzene			37 - 162
2-Hexanone			NS
Methylene chloride			D - 221
4-Methyl-2-pentanone			NS
Styrene			NS
Tetrachloroethene			64 - 148
1,1,2,2-Tetrachloroethane			46 - 157
Toluene	47 - 150	≤53	47 - 150
1,1,1-Trichloroethane			52 - 162
1,1,2-Trichloroethane			52 - 150
Trichloroethene	71 - 157	≤50	71 - 157
Vinyl acetate			NS
Vinyl chloride			D - 251
Xylenes			NS
Surrogates			
1,4-Bromofluorobenzene	86 - 115	NA	86 - 115[c], 74 - 121[d]
1,2-Dichloroethane-d_4	76 - 114	NA	76 - 114[c], 70 - 121[d]
Toluene-d_8	88 - 110	NA	88 - 110[c], 81 - 117[d]

[a]All limits are based on a certain spike concentration. If this concentration changes, the limits also change. See Tables 6 and 7 of SW8240.

[b]Method specified limits from Chapter One, SW-846 and tables in method. MS/MSD results used for post-analysis, project review; LCS results used to control analytical system.

[c]Aqueous matrix limits.

[d]Solid matrix limits.

NA = Not applicable.
RPD = Relative percent difference.
NS = Not specified.
LCS = Laboratory control sample.

Table 1.10-6
Quality Control Acceptance Criteria for Volatile Organic Compounds by Method SW8260[a]

Parameter	Liquid Matrix Spike		LCS Spike
	Accuracy[b] (% Recovery)	Precision (RPD)	Accuracy[b] (% Recovery)
Acetone			3-127
Benzene	74-132	≤60	74-132
Bromodichloromethane			64-132
Bromoform			41-135
Bromomethane			46-152
2-Butanone (MEK)			D-160
Carbon disulfide			29-223
Carbon tetrachloride			53-167
Chlorobenzene	73-119	≤14	73-119
Chloroethane			50-154
Chloroform			64-130
Chloromethane			39-135
Dibromochloromethane			60-122
Dichlorodifluoromethane (Freon 12)			5-157
1,1-Dichloroethane			65-131
1,2-Dichloroethane			68-138
1,1-Dichloroethene	51-133	≤23	51-133
trans-1,2-Dichloroethene			58-144
1,2-Dichloropropane			77-119
trans-1,3-Dichloropropene			64-132
cis-1,3-Dichloropropene			75-131
Ethylbenzene			72-130
2-Hexanone			58-140
Methylene chloride			49-151
4-Methyl-2-pentanone			58-142
Styrene			73-131
Tetrachloroethene			62-124
1,1,2,2-Tetrachloroethane			60-134
Toluene	81-121	≤14	81-121
1,1,1-Trichloroethane			58-144
1,1,2-Trichloroethane			68-122
Trichloroethene	73-117	≤13	73-117
Trichlorofluoromethane (Freon 11)			50-142
Vinyl acetate			35-199
Vinyl chloride			27-161
Xylene; m/p-			74-128
Xylene; o-			79-125
Surrogates[b]			
1,4-Bromofluorobenzene	84-116 (86-115)	NA	84-116 (86-115)
1,2-Dichloroethane-d_4	83-121	NA	83-121
Toluene-d_8	81-115 (88-110)	NA	81-115 (88-110)

[a] Method 8260 does not list tolerances for LCS or MS recoveries. The tolerances were derived from historical data from Radian laboratory.

[b] Derived form historical data from Radian laboratory. Method 8260 tolerances shown in parentheses.

NA = Not applicable.
RPD = Relative percent difference.
NS = Not specified.
LCS = Laboratory control sample.

Table 1.10-7
Quality Control Acceptance Criteria for Semivolatile Organic Compounds by Method SW8270

Parameter	Aqueous Matrix Spike		Solid Matrix Spike		LCS Spike
	Accuracy[a] (% Recovery)	Precision (RPD)	Accuracy[a] (% Recovery)	Precision (RPD)	Accuracy[a] (% Recovery)
Base/Neutral Extractables					
Acenaphthene	47 - 145	≤56	47 - 145	≤56	47 - 145
Acenaphthylene					33 - 145
Anthracene					27 - 133
Benzo(a)anthracene					33 - 143
Benzo(b)fluoranthene					24 - 159
Benzo(k)fluoranthene					11 - 162
Benzo(ghi)perylene					D - 219
Benzo(a)pyrene					17 - 163
Benzyl alcohol					NS
bis(2-Chloroethoxy)methane					33 - 184
bis(2-Chloroethyl)ether					12 - 158
bis(2-Chloroisopropyl)ether					36 - 166
bis(2-Ethylhexyl)phthalate					8 - 158
4-Bromophenyl phenyl ether					53 - 127
Butyl benzyl phthalate					D - 152
4-Chloroaniline					55-153[b,d]/59-163[c,d]
2-Chloronaphthalene					60 - 118
4-Chlorophenyl phenyl ether					25 - 158
Chrysene					17 - 168
Dibenzo(a,h)anthracene					D - 227
Dibenzofuran					67-122[b,d]/67-126[c,d]
Di-n-butylphthalate					1 - 118
1,2-Dichlorobenzene					32 - 129
1,3-Dichlorobenzene					D - 172
1,4-Dichlorobenzene	20 - 124	≤81	20 - 124	≤81	20 - 124
3,3'-Dichlorobenzidine					D - 262
Diethyl phthalate					20-162[b,d]/67-143[c,d]
Dimethyl phthalate					D-179[b,d]/68-127[c,d]
2,4-Dinitrotoluene	39 - 139	≤62	39 - 139	≤62	39 - 139
2,6-Dinitrotoluene					50 - 158
Di-n-octylphthalate					4 - 146
Fluoranthene					26 - 137
Fluorene					59 - 121
Hexachlorobenzene					D - 152
Hexachlorobutadiene					23-140[b,d]/40-137[c,d]
Hexachlorocyclopentadiene					0-308[b,d]/0-249[c,d]
Hexachloroethane					42-165[b,d]/53-143[c,d]
Indeno(1,2,3-cd)pyrene					D - 171
Isophorone					21 - 196
2-Methylnaphthalene					37-150[b,d]/30-168[c,d]
Naphthalene					21 - 133
2-Nitroaniline					40-149[b,d]/28-167[c,d]
3-Nitroaniline					45-157[b,d]/60-152[c,d]
4-Nitroaniline					25-162[b,d]/42-155[c,d]
Nitrobenzene					35 - 180
n-Nitrosodiphenylamine					NS
n-Nitrosodipropylamine	D - 230	≤113	D - 230	≤113	D - 230
Phenanthrene					54 - 120
Pyrene	52 - 115	≤43	52 - 115	≤43	52 - 115
1,2,4-Trichlorobenzene	44 - 142	≤59	44 - 142	≤59	44 - 142

Table 1.10-7
(Continued)

Parameter	Aqueous Matrix Spike		Solid Matrix Spike		LCS Spike
	Accuracy[a] (% Recovery)	Precision (RPD)	Accuracy[a] (% Recovery)	Precision (RPD)	Accuracy[a] (% Recovery)
Acid Extractables					
Benzoic acid					0-294[b,d]/0-197[c,d]
4-Chloro-3-methylphenol	22 - 147	≤84	22 - 147	≤84	22 - 147
2-Chlorophenol	23 - 134	≤81	23 - 134	≤81	23 - 134
2,4-Dichlorophenol					39 - 135
2,4-Dimethylphenol					D-112[b,d]/D-116[c,d]
4,6-Dinitro-2-methylphenol					D - 181
2,4-Dinitrophenol					D - 191
2-Methylphenol					29-133[b,d]/33-132[c,d]
4-Methylphenol					20-135[b,d]/25-135[c,d]
2-Nitrophenol					29 - 182
4-Nitrophenol	D - 132	≤124	D - 132	≤124	D - 132
Pentachlorophenol	14 - 176	≤86	14 - 176	≤86	14 - 176
Phenol	5 - 112	≤76	5 - 112	≤76	5 - 112
2,4,5-Trichlorophenol					37-121[b,d]/61-116[c,d]
2,4,6-Trichlorophenol					37 - 144
Surrogates					
2-Fluorobiphenyl	43 - 116	NA	30 - 115	NA	43-116[b], 30-115[c]
2-Fluorophenol	21 - 139	NA	32 - 132	NA	21-139[b,d], 32-132[c,d]
Nitrobenzene-d_5	35 - 114	NA	23 - 120	NA	35-114[b], 23-120[c]
Phenol-d_5	4 - 162	NA	48 - 127	NA	4-162[b,d], 48-127[c,d]
Terphenyl-d_{14}	33 - 141	NA	18 - 137	NA	33-141[b], 18-137[c]
2,4,6-Tribromophenol	10 - 123	NA	19 - 122	NA	10-123[b], 19-122[c]

[a]Method specified limits from Chapter One, SW-846 and tables in method. MS/MSD results used for post-analysis, project review; LCS results are used for control of analytical system.

[b]Aqueous matrix limits

[c]Solid matrix limits.

[d]Laboratory derived limits.

D = Detected.
NA = Not applicable.
NS = Not specified.
RPD = Relative percent difference.
LCS = Laboratory control sample.

Table 1.10-8
Quality Control Acceptance Criteria for Polychlorinated Dioxins and Furans by Method SW8280

Parameter	LCS Spike	
	Accuracy[a] (% Recovery)	Precision (RPD)
2,3,7,8-TCDD	66-140	21
Surrogates		
C_{13}-2,3,7,8-TCDD	40-120	50
C_{13}-TCDD	40-120	50
C_{13}-TCDF	40-120	50
C_{13}-PeCDD	40-120	50
C_{13}-PeCDF	40-120	50
C_{13}-HxCDD	40-120	50
C_{13}-HxCDF	40-120	50
C_{13}-HpCDD	40-120	50
C_{13}-HpCDF	40-120	50
C_{13}-OCDD	40-120	50
C_{13}-OCDF	40-120	50

[a]Method specified limits from Chapter One, SW-846 and tables in method. Duplicate results used for post-analysis, project review; LCS results are used for control of analytical system.

[b]Aqueous matrix limits

[c]Solid matrix limits.

[d]Laboratory derived limits.

D = Detected.
NA = Not applicable.
NS = Not specified.
RPD = Relative percent difference.
LCS = Laboratory control sample.

- Maintenance responsibilities;
- Maintenance schedules for major and/or critical instrumentation and apparatus; and
- An adequate inventory of critical spare parts and equipment.

These areas are discussed in the following subsections.

1.12.1 Procedures

Equipment and apparatus used in environmental measurement program falls into two general categories:

- Equipment permanently assigned to a specific laboratory (e.g., GC laboratory, GC/MS laboratory, etc.); and
- Field sampling equipment available for use on an as-needed basis (e.g., field meters, pumps, vehicles, etc.).

Maintenance responsibilities for laboratory instruments are assigned to the respective laboratory managers. The laboratory managers then establish maintenance procedures and schedules for each major equipment item. This responsibility may be delegated to laboratory personnel, although the managers retain responsibility for ensuring adherence to prescribed protocol. All laboratories are bound by analytical contractual agreements to maintain the ability to produce data that meet the project objectives and to follow method specifications. This ensures that adequate spare parts, maintenance, schedules, and emergency repair services are available.

Maintenance responsibilities for field equipment are assigned to the project director and task leaders for specific sampling tasks. However, the field team using the equipment is responsible for checking the status of the equipment prior to use, and reporting any problems encountered. The field team is also responsible for ensuring that critical spare parts are included as part of the field equipment. Critical spare parts are included in the field equipment checklist.

1.12.2 Maintenance Schedules

The effectiveness of any maintenance program depends to a large extent on adherence to specific maintenance schedules for each major equipment item. Other maintenance activities are conducted on an as-needed basis. Manufacturers' recommendations provide the primary basis for the established maintenance schedules, and manufacturers' service contracts provide primary maintenance for many major instruments (e.g., GC/MS instruments, atomic absorption spectrometers, analytical balances, etc.). Maintenance activities are documented in a maintenance log, which indicates the required frequency for each procedure and also provides for dated entries.

1.12.3 Spare Parts

Along with a schedule for maintenance activities, an adequate inventory of spare parts is required to minimize equipment down time. The inventory includes those parts (and supplies) that:

- Are subject to frequent failure;
- Have limited useful lifetimes; or
- Cannot be obtained in a timely manner should failure occur.

Field sampling task leaders and the respective laboratory managers are responsible for maintaining an adequate inventory of spare parts. In addition to spare parts and supply inventories, nonassigned equipment (equipment that is not permanently assigned to any one project) represents an in-house source of backup equipment and instrumentation.

1.13 Procedures Used to Assess Data Precision, Accuracy, and Completeness

The evaluation/assessment of measurement data is required to ensure that the QA objectives for the program are met and that quantitative measures of data quality are provided. The data evaluation proce-

dures, calculations and applications used for the Galena Airport RI/FS are based on the U.S. EPA *Guidelines for Assessing and Reporting Data Quality for Environmental Measurements* (January 1983).

A distinction must be made between routine QC and data assessment that is conducted as a part of laboratory operations, and the project-related data assessment process conducted after the data have been reported. It must be assumed that the planning, standard procedures, and monitoring activities conducted during the sampling and analysis process have served to control the process as much as possible to produce data of sufficient quality for project needs. After the data have been reported, it is necessary to identify any part of the process that could not be controlled, and to what extent that may affect the quality of the reported data.

The routine QC procedures conducted in the laboratory are established in the published methods, this document, and the analytical SOPs. The laboratory is responsible for following those procedures and operating the analytical systems within statistical control limits. These procedures include proper instrument maintenance, calibration and calibration checks, and internal QC sample analyses at the required frequencies (i.e., reagent blanks, surrogate spikes, matrix spike/matrix spike duplicate [MS/MSD], analytical spikes, laboratory duplicates). One of the additional ongoing data assessment processes is maintaining control charts for representative QC sample analyses to monitor system performance. This provides verification that the system is in statistical control, and indicates when performance problems occur, so the problems can be corrected as soon as possible. When reporting the sample data, the laboratory is required to provide the results of associated QC sample analyses so the project staff can evaluate the performance of the analytical process.

Problems with analytical data sometimes occur in spite of all precautions taken in planning and execution of the sampling and analysis task. In these cases, the data assessment conducted by the project QA staff after the data have been reported must identify the problem, determine which data are affected, state how these data may be limited for use in the intended applications, and make recommendations for corrective actions as necessary. If the Galena Airport scope is changed, then a review may be provided.

The discussion of data assessment presented in this section pertains to the project-related assessment of data that may be performed *after* data have been reported and laboratory analyses have been completed.

Data assessment procedures that could be performed for the Galena Airport RI/FS include:

- Initial review of analytical and field data for complete and accurate documentation, holding time compliance, and required frequency of QC samples;
- Evaluation of blank results to identify systematic contamination;
- Statistical calculations for accuracy and precision using the appropriate QC sample results;
- Estimates of completeness, in terms of the percent of valid unqualified data; and
- Assigning data qualifier flags to the data as necessary to reflect limitations identified by the process.

Qualified data would be discussed in the task reports, and data flags could be transmitted to users via data tables from the database and in analytical data reports.

1.13.1 Formulas

Several of the data validation acceptance criteria involve specific calculations. Example calculations are presented below.

1.13.1.1 Instrument Response Linearity (Calibration)

Acceptance criteria for instrument response

linearity checks are based upon the correlation coefficient, r, of the best fit line for the calibration data points. The correlation coefficient reflects the linearity of response to the calibration standards and is calculated as:

$$r = \frac{n\sum(xy) - (\sum x)(\sum y)}{\sqrt{[n\sum x^2) - (\sum x)^2][n\sum y^2) - (\sum y)^2]}}$$

where: x = calibration concentrations;
y = instrument response (peak area); and
n = number of calibration points (x,y data pairs).

1.13.1.2 Precision

Control limits for control sample analyses, acceptability limits for replicate analyses, and response factor agreement criteria specified for calibration and internal QC checks are based upon precision, in terms of the coefficient of variation (CV) or the relative percent difference (RPD). The standard deviation of a sample set is calculated as:

$$S = \textit{standard deviation} = \sqrt{\frac{\sum (x-\bar{x})^2}{(n-1)}}$$

where: x = individual measurement;
$\bar{x}$ = mean value for the individual measurements; and
n = number of measurements.

The CV is then calculated as:

$$CV = \left(\frac{S}{\bar{x}}\right) x\ 100\%$$

The RPD calculation allows for the comparison of two analysis values in terms of precision with no estimate of accuracy. RPD is calculated as:

$$RPD = \frac{|M-m|}{\left(\frac{M+m}{2}\right)} x\ 100\%$$

where: M = first measurement value; and
m = second measurement value.

For duplicate measurements, CV is related to RPD by the following:

$$CV = \frac{RPD}{\sqrt{2}}$$

1.13.1.3 Accuracy

The accuracy of data is typically summarized in terms of relative error (RE). This calculation reflects the degree to which the measured value agrees with the actual value, in terms of percent of the actual value. Relative error is calculated as:

$$\%\ \textit{Relative Error} = \frac{\textit{Measured Value} - \textit{Actual Value}}{\textit{Actual Value}} x\ 100$$

This way of expressing accuracy allows for a comparison of accuracy at different levels (e.g., different concentrations), and for different parameters of the same type (e.g., different compounds analyzed by the same method). Control sample analyses are typically evaluated using this calculation.

In this program, another calculation is frequently used to assess the accuracy of a procedure. Percent recovery is a calculation used to determine the performance of many of the QC checks. Percent recovery is calculated as:

$$\%\ \textit{Recovery} = \frac{\textit{Measured Value}}{\textit{Actual Value}} x\ 100$$

Another similar calculation used to determine the performance of a method for recovery of a spike concentration added to a sample is the percent

spike recovery calculation. The percent spike recovery is determined as:

$$\% \text{ Spike Recovery} = \frac{(\text{Value of Sample Plus Spike}) - (\text{Value of Unspiked Sample})}{(\text{Value of Spike Added})} \times 100$$

1.13.2 Control Limits

Control limits for central tendency and variability are generated by the laboratory to statistically monitor system performance. These limits are within method specified tolerances. Since control limits may change as the analytical system is improved and matrices change, these limits are not provided in this plan.

1.13.3 Documentation

Data reviewed to perform each of the above procedures and the implications to natural sample results are discussed in each of the following subsections.

1.13.3.1 Blank Data Assessment

Reagent blank results indicate whether any of the contaminants reported in sample results may be attributed to laboratory sources (reagents, glassware, instrumentation) and were not likely present in the sampled medium. The most common laboratory contaminants are methylene chloride, phthalates, acetone, and toluene; these are recognized as being ubiquitous in the laboratory environment and controlling them to within acceptable low levels is part of standard laboratory procedures.

If contamination from these compounds is reported in reagent blanks, the samples associated with the blank, either the same analytical or extraction batch, may be qualified to indicate that some or all of these compounds may be from laboratory sources. If the concentrations reported in the samples are similar to the blank concentrations, it is likely that all of the contamination was introduced, and this assessment is made in the QA/QC report for the sampling task.

In some cases, where there is a large sampling task and reagent blank results indicate a more significant contamination problem, a more systematic approach may be applied. This approach is only used when a series of reagent blanks analyzed over a period of time are reported. The assessment criterion is calculated from reagent blank results as the mean concentration plus three standard deviations for each contaminant reported. The sample data are assessed using this criterion. Sample concentrations below the criterion are considered to be most likely from laboratory sources, and at least some of the sample concentrations higher than that are considered to be from the sampled medium (aquifer, soil etc.). This semi-quantitative approach is used only as a tool to screen the sample results and provide a common basis for further assessment; none of the results are censored or changed in any way in the database or for reporting. The assessment is discussed in the QA report for the sampling task. Samples with blank contamination problems will be assigned a data qualifier flag.

Results for other types of blanks such as equipment, ambient, or trip blanks are assessed individually. The probable source of contamination is identified, and the associated sample results are qualified as necessary. For example, if equipment blank results show contamination, and the sample collected from the bailer shows the same compound, the sample results will be qualified to indicate the probable level of introduced contamination.

1.13.3.2 Accuracy

As previously defined, accuracy is associated with correctness, and is a comparison between a measured value and a known, or "true" value. Accuracy is calculated from method spike (spikes of the pure matrix) matrix spike, or LCS results.

Spike results are reported by the laboratory as percent recovery and are compared to the accuracy objectives stated in section 1.4. Results that do not satisfy the objectives are assigned a data qualifier flag to indicate uncertainty associated with inaccuracy.

Method spikes are spikes of a reference material in a water matrix. If recovery is outside the established limits, samples from the same extraction batch may be qualified. Matrix spike results are generally more sample specific. If matrix spike

recovery is outside the established limits, results for samples collected from similar conditions and/or handled in the same batch will be examined. If any results appear atypical and could be related, those results may also be qualified. The flagged data will be discussed in the QA/QC report for the sampling task, and specific limitations such as poor or enhanced recovery for specific compounds will be stated. Further investigation or corrective action may be taken to find methods to reduce the interferences.

Surrogate spike results are also reported and used to assess recovery of target analytes on a sample by sample basis and provide a measure of system performance. Surrogate spike recoveries are compared to recovery limits. Any results outside the limits are flagged on laboratory reports and in the database. Any corrective action taken in the laboratory is documented in laboratory performance records and/or discussed in the comment section of the data report.

Confidence intervals can be calculated for an analytical method if performance evaluation samples are submitted or a series of method spikes is analyzed. The results are used to define confidence intervals for the recovery of each compound analyzed.

1.13.3.3 Precision

Precision is a measure of variability between duplicate or replicate analyses, and is calculated for field and laboratory replicates. By definition, field or total precision incorporates laboratory precision. Precision is calculated as the RPD between duplicate samples or analyses, or matrix spike/matrix spike duplicates as appropriate. The calculated RPDs are compared to the objectives stated in section 1.4. Results that do not satisfy the objectives are assigned a data qualifier flag indicating uncertainty associated with imprecision.

An average RPD may be calculated and reported as a measure of overall analytical precision for compounds with multiple measurements. The specific samples collected or analyzed in duplicate are flagged if they do not satisfy the QA objectives. In addition, associated samples may be flagged to indicate variability from precision. For poor field duplicate precision, samples collected by the same sampling team, from the same equipment, or on the same day may be affected. Close evaluation of those results should indicate the most likely source of variability, and the corresponding samples will be qualified as warranted. For poor laboratory precision, samples processed and analyzed in the same batch will be more closely evaluated, and any anomalous results will be qualified.

The QA coordinator is responsible for ensuring that data qualifier flags are assigned to the data as required by the established QC criteria, and that they are reported and understood by project staff using the data for specific applications. The QA coordinator is also responsible for initiating corrective actions for analytical problems identified during the QC data assessment process. These corrective actions range from verifying that the method was in statistical control during the analytical runs, to re-analysis of the sample, or resampling.

1.13.3.4 Completeness

Completeness is calculated after the QC data have been evaluated, and the results applied to the measurement data. In addition to results identified as being outside of the QC limits established for the method, broken or spilled samples, or samples that could not be analyzed for any other reason are included in the assessment of completeness. The percentage of valid results is reported as completeness.

For the Galena Airport project, completeness will be calculated as follows:

$$\frac{T - (I-NC)}{T} \times 100\% = Completeness.$$

where: T = Total number of expected measurements for a method and matrix;
I = Number of invalidated results for a method and matrix; and
NC = Number of results not collected (e.g., bottles broken etc.) for a method and a matrix.

1.14 Corrective Action

During the course of the Galena Airport RI/FS, it is the responsibility of the project director, task leaders, QA coordinator, and sampling team members to ensure that all measurement procedures are followed as specified and that measurement data meet the prescribed acceptance criteria presented in tables of section 1.10. In the event a problem arises, it is imperative that prompt action be taken to correct the problem.

1.14.1 Corrective Action Response

The on-site QA coordinator, task leaders, or other project members will initiate a corrective action request in the event that QC results exceed acceptability limits, or upon identification of some other problem or potential problem. Method specified responses are presented in section 1.10, Tables 1.10-9 through 1.10-16. Such problems are followed up by the technical director or QA officer. Corrective action is also initiated by the QA Coordinator based upon QC data or audit results. Corrective actions range from use of data qualifier flags, to reanalysis of the sample or samples affected, to resampling and reanalysis, to recommending a change in procedures, depending upon the severity of the problem. Problems that require corrective action are documented by the use of a Corrective Action Report (CAR), as presented in Figure 1.14-1.

1.14.2 Reestablishment of Control

Procedures for reevaluation and reestablishment of control are summarized in section 1.10, Tables 1.10-9 through 1.10-16. Generally, the analytical laboratory chosen will be expected to follow method specified procedures.

1.14.3 Documentation for Corrective Action

A system for issuing formal Recommendations for Corrective Action (RCAs) exists for addressing problems signaling significant and systematic deficiencies identified through independent QA review. Recommendations for corrective actions are issued only by a member of the QA Group, or a designee in a specific QA role. Each RCA addresses a specific problem or deficiency, usually identified during QA audits of laboratory or project operation. An example RCA form is presented as Figure 1.14-2. Each of these formal written recommendations requires a written response from the responsible party (i.e., to whom the RCA was issued). A summary of the "unresolved" RCAs is prepared by the QA group on a monthly basis and issued to laboratory management. These reports list all RCAs that have been issued, the manager responsible for the work area, and the current status of each RCA. Each RCA requires a response and verification by the QA group that the corrective action has been implemented before the status is changed on the monthly report. In the event that there is no response to an RCA within 30 days, or the proposed corrective action is disputed, the recommendation and/or conflict is pursued to successively higher management levels until the issue is resolved. The corrective action scheme is shown in the form of a flow chart in Figure 1.14-3.

1.15 Quality Assurance

The QA coordinator and QC task members will issue QA reports to the project management, task leaders, and laboratory supervisors describing the results of QC measurements, performance audits, and systems audits performed for each sampling and analysis task. Audit results will be summarized in the reports; detailed audit results and checklists will be submitted according to the procedures described in section 1.11.

1.15.1 Reporting Procedure

One QA report is planned for the Galena Airport project. This report will be issued with the ITIR and will publish results of data validation and evaluation tasks.

1.15.2 Report Content

The content for the QA Reports with included the following topics:

- Summary of sampling and analytical activity and highlights of QA results;
- Measurement data accuracy, precision, and completeness (per sample matrix and method); and

Table 1.10-9
Summary of Calibration and Internal Quality Control for Indicator Parameters

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]
ASTM D2216 (modified)	Moisture	Balance calibration	Daily	0.01g	Service balance
		Duplicate analysis	10%	RPD < 30%	Obtain third value; flag data
AK101	Gasoline Range Organics	Multipoint calibration (minimum 3 points)	Initial caibration prior to sample analysis, or when daily calibration verification fails	%RSD <25	Identify and repeat outlying point(s); recalculate curve using repeated point(s)
		Initial and continuing calibration verification (ICV and CCV)	Daily prior to sample analysis, and once every 12 hours	RF ≤25% of calibration curve	1) Repeat CCV 2) If still out, recalibrate
		Second source laboratory control standard (LCS)	Once every 10 samples, and at the end of each sequence	±25% of the true value	1) Reanalyze LCS, if still out correct problem 2) Reanalyze associated samples
		Surrogate control sample (SCS)	Once every 20 samples or one per batch	60-120% recovery	1) Reanalyze SCS 2) If LCS is acceptable, flag data 3) If LCS is unacceptable, reanalyze associated samples
		Retention time (RT) window	One 72-hour study performed on each GC column and whenever a new column is installed	Anually	1) Perform maintenance 2) Repeat test
		Matrix spike (MS) and matrix spike duplicate (MSD); level of spike should be approximately 100-250 times the detection limit	1 MS and 1 MSD per every 20 Air Force project samples	60-120% recovery	1) Analyze a LCS containing each analyte that failed criteria 2) If recovery for LCS is outside of designated range, the system is considered to be out of control. Immediate identification and correction of problem is required. 3) Reanlyze any samples affected by out-of-control condition 4) If LCS is within criteria, flag data

Table 1.10-9
(Continued)

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]
AK101 continued		Reagent blank	Once per analytical batch and after highly contaminated samples	< MRL	1) Source of contamination investigated. 2) Appropriate corrective action taken and documented 3) All samples processed with a contaminated blank are to be re-extracted and re-analyzed at no cost to the Air Force if still within HT; otherwise, notify QA officer for decision. 4) Sample results associated with reagent blank contamination at ≤3 times the detection limit are to be flagged.
		Surrogate	Every sample, spike, standard, and reagent blank	50-150% recovery	1) Recalculate results 2) If still out, reanalyze sample 3) If still out, flag result and document in report that steps 1 and 2 were performed.
AK102	Diesel Range Organics	Multipoint calibration	Initial caibration prior to sample analysis, or when daily calibration verification fails	%RSD <25	Identify and repeat outlying point(s); recalculate curve using repeated point(s)
		Initial and continuing calibration verification (ICV and CCV)	Daily prior to sample analysis, and every 12 hours	RF ≤25% of calibration curve	1) Repeat CCV 2) If still out, recalibrate
		Second source laboratory control standard (LCS)	Once every 10 samples, and at the end of each sequence	±25% of the true value	1) Reanalyze LCS, if still out correct problem 2) Reanalyze associated samples
		Surrogate control sample (SCS)	Once every 20 samples or one per batch	60-120% recovery	1) Reanalyze SCS 2) If LCS is acceptable, flag data 3) If LCS is unacceptable, reanalyze associated samples
		Retention time (RT) window	One 72-hour study performed on each GC column and whenever a new column is installed	Anually	1) Perform maintenance 2) Repeat test

Table 1.10-9
(Continued)

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action*
AK102 continued		Matrix spike (MS) and matrix spike duplicate (MSD); level of spike should be approximately 100-250 times the detection limit	1 MS and 1 MSD per every 20 Air Force project samples	60-120% recovery	1) Analyze a LCS containing each analyte that failed criteria 2) If recovery for LCS is outside of designated range, the system is considered to be out of control. Immediate identification and correction of problem is required. 3) Reanlyze any samples affected by out-of-control condition 4) If LCS is within criteria, flag data
		Reagent blank	Once per analytical batch and after highly *contaminated* samples	< MRL	1) Source of contamination investigated. 2) Appropriate corrective action taken and documented 3) All samples processed with a contaminated blank are to be re-extracted and re-analyzed at no cost to the Air Force if still within HT; otherwise, notify QA officer for decision. 4) Sample results associated with reagent blank contamination at ≤3 times the detection limit are to be flagged.
		Surrogate	Every sample, spike, standard, and reagent blank	50-150% recovery	1) Recalculate results 2) If still out, reanalyze sample 3) If still out, flag result and document in report that steps 1 and 2 were performed.

*All corrective actions associated with Air Force project work shall be documented and the records maintained by the laboratory as specified in the IRP Handbook.

NA = Not applicable.

Table 1.10-10
Summary of Calibration and Internal Quality Control Procedures for SW7060, SW7421 and SW6010

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]
SW7060 SW7421	Arsenic *Lead* Refer to: SW846-3020[a] SW7060[a] SW7421[a] IRP Handbook[b]	Initial Calibration	Daily before any other analysis	Correlation coefficient ≥0.995	Identify and repeat outlying point(s); recalculate curve using repeated points
		Initial Calibration Verification	Daily before batch one is analyzed	90 - 110% of theoretical value	Accuracy: 1. Repeat calibration verification 2. If still out, identify and correct problem, run calibration verification again;if still out, recalibrate
		Method Blank	Once per batch	Measured concentrations for all analytes must be <PRDL or 20x the concentration of the lowest sample	1. Determine source of contamination 2. Redigest samples in the preparation batch
		LCS/LCSD	One LCS/LCSD pair per batch	1. Accuracy: 75-125% 2. Precision: 20% RPD	1. Accuracy: Analyze a third LCS. If recovery for same analytes are still out, stop, identify, and correct problem before proceeding 2. Precision: Analyze a third LCS. If recovery for same analytes are still out, stop, identify, and correct problem before proceeding
		MS/MSD	One MS/MSD pair per batch	1. Accuracy: 75-125% 2. Precision: 20% RPD	1. Accuracy: a. Reanalyze spiked sample b. If still out, and LCS/LCSD results are within acceptable limits, flag MS/MSD results as matrix interference 1. Precision: a. Reanalyze spiked sample b. If still out, and LCS/LCSD results are within acceptable limits, flag MS/MSD results as matrix interference

Table 1.10-10
(Continued)

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]
SW7060 SW7421 con't		Continuing Calibration Verification	After 10th sample and at the end of the batch	80-120% of true value	Accuracy: 1. Repeat calibration verification 2. If still out, identify and correct problem, then reanalyze all samples analyzed since last valid calibration verification
		Serial Dilution	Once per batch	10% agreement with undiluted value if undiluted value >25 x MDL	Perform analytical spike
		Analytical Spike	As corrective action for seri. dilution	85-115% recovery	Perform method of standard additions
SW6010	ICP M.. als Refer to: SW846-3005[a] SW846-3050[a] SW6010[a] IRP Handbook[b]	Initial calibration standard as per SW846-6010 (2 points)	Daily, prior to sample analysis	NA	1) Correct problem according to instrument manufacturer's *recommendations* 2) Repeat calibration
		Initial Calibration Verification (ICV) (concentration = upscale calibration standard)	Following initial calibration	*Within ±15% of expected* value	1) Repeat ICV 2) Recalibrate if still out
		Continuing calibration verification (CCV), single point	Every 10 samples and at end of analytical run	Within ±10% of expected value	1) Repeat CCV 2) Locate and correct problem 3) Recalibrate if still out 4) Reanalyze associated samples
		Calibration blank	Every 10 samples and at end of analytical run	< Quantitation limits	1) Perform system blank 2) If system blank is contaminated, identify and correct source of contamination, then repeat calibration blank analysis 3) Reanalyze previous 10 samples
		Liquid, digested, second source Laboratory Control Sample (LCS)	Daily (as per Instrument Calibration requirements in IRP Handbook [b])	80-120% recovery	1) Analyze third LCS, if still out correct problem 2) Reanalyze associated samples
		Interference check sample	Run at the more frequent of the following: 1) Beginning and end of analytical run; or 2) Twice during every 8-hour work shift	Within ±20% of expected value for instrument check standard elements	1) Correct source of interference and rerun sample 2) See Lab Manager if still out

Table 1.10-10
(Continued)

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action[a]
SW6010 (Cont'd)		Method blank	Once per day/digestion batch	< Method reporting limits unless the lowest concentration of analyte in the samples is 20x the blank concentration	1) Investigate sources of contamination 2) Appropriate corrective action taken and documented 3) All samples processed with a contaminated blank are to be redigested and reanalyzed at no cost to the Air Force
		Matrix spike (MS)/matrix spike duplicate (MSD) (portion of sample is spiked prior to digestion)	1 MS and 1 MSD per every 20 Air Force project samples	75-125% recovery; RPD = ±20%	Accuracy 1) Reanalyze spiked sample 2) If still out and LCS and/or LCSD within acceptance criteria, then flag MS/MSD as matrix interference Precision 1) Reanalyze spiked sample 2) If still out and LCS and/or LCSD within acceptance criteria, then flag MS/MSD as matrix interference
		MDL Study	Once per year	Detection limits established shall not exceed those in Table 2-1 of IRP Handbook[b]	Detection limits which exceed established criteria shall be submitted to the Air Force for approval prior to analysis of any project samples[b]
		Linear Range Study	Once per year	NA	NA

[a]Test Methods for Evaluating Solid Waste, U.S. EPA, SW846, September 1986.

[b]IRP Handbook, May 1991.

[c]All corrective actions associated with Air Force project work shall be documented and the records maintained by the laboratory, as specified in the IRP Handbook.

NA = Not Applicable.

Table 1.10-11
Summary of Calibration and Internal Quality Control Procedures for SW8080

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action[c]
SW8080	Organochlorine pesticides, and PCBs Refer to: SW846-3500[a] SW846-3600[a] SW846-8000[a] IRP Handbook[b]	Five-point calibration (for all analytes)	Initial calibration prior to sample analysis	a) RSD < %20 for RFs or r $\geq$0.995	Identify and repeat outlying point(s); recalculate curve using repeated points
		Initial daily Calibration Verification (ICV)	Daily, before sample analysis	Response for any analyte within $\pm$15% of predicted response for primary column; $\pm$20% for confirmation column	1) Repeat ICV 2) If still out, identify and correct problem before proceeding 3) Recalibrate if necessary
		Retention time (RT) windows calculated for each analyte	One 72-hour study performed on each GC column and whenever a new column is installed	See Method 8000	1) Perform maintenance 2) Repeat test
		Second source Laboratory Control Sample (LCS) see Section 10.0 tables for list of analytes spiked	Daily (as per Instrument Calibration requirements in IRP Handbook[b]). Must be run prior to samples.	Recovery for all analytes within QC acceptance criteria Table 1.10-3	1) Repeat LCS 2) If still out, stop, identify and correct problem before proceeding with samples
		Continuing Calibration Verification (CCV)	10% sample frequency, minimum of two per set	a) Primary: Recovery of analytes within $\pm$15% of daily calibration factor cinfirmation: $\pm$20% of daily calibration factor b) Ongoing calibration analytes elute within daily RT windows	1) Locate and correct source of problem--document actions taken 2) Repeat test only for analytes that failed to meet criteria 3) Repeat test for all compounds of interest
		Breakdown check (Endrin and DDT)	Daily prior to analysis of samples and as needed during analysis	Degradation $\leq$20% (each)	Perform system maintenance as per SW846-8000
		Matrix spike (MS) and matrix spike duplicate (MSD); level of spike should be approximately 100-250 times the detection limit	1 MS and 1 MSD per every 20 Air Force project samples	QC Acceptance Criteria Table 1.10-3.	1) Analyze a LCS containing each analyte that failed criteria 2) If recovery for LCS is outside of designated range, the system is considered to be out of control. Immediate identification and correction of problem is required. 3) Reanlyze any samples affected by out-of-control condition 4) If LCS is within criteria, flag data
		Two surrogate standards spiked into each sample. Dibutylchlorendate (DBC) is primary surrogate	Every sample, spike, standard, and reagent blank	Table 1.10-3. Only one surrogate must meet acceptance criteria.	1) Recalculate results 2) If possible reextract and reanalyze 3) If 2 not possible, reanalyze and flag results

Table 1.10-11
(Continued)

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action[c]
SW8080 (Cont'd)		Method blank	One per extraction batch per instrument	< Method reporting limits	1) Source of contamination investigated. 2) Appropriate corrective action taken and documented 3) All samples processed with a contaminated blank are to be re-extracted and re-analyzed at no cost to the Air Force if still within HT; otherwise, notify QA officer for decision. 4) Sample results associated with method blank contamination are to be flagged.
		Second-column confirmation, to meet IRP requirements[b]	50% for all positive results above the reporting limit	Analysis of standards and samples performed on a second GC column of dissimilar phase and retention characteristics (or GC/MS if concentration detected is sufficient) within specified holding times	Resampling and reanalysis performed at no cost to government, even if first column analysis was conducted within holding time[b]
		MDL study	Once per year	Detection limits established shall not exceed those in Table 2-1 of IRP Handbook[b]	Detection limits that exceed estab-lished criteria shall be submitted to the Air Force for approval prior to the analysis of any project samples[b]

[a]Test Methods for Evaluating Solid Waste, U.S. EPA, SW846, September 1986.

[b]IRP Handbook, May 1991.

[c]All corrective actions associated with Air Force project work shall be documented and the records maintained by the laboratory, as specified in the IRP Handbook.

NA = Not applicable.

Table 1.10-12
Summary of Calibration and Internal Quality Control Procedures for SW8240

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action[c]
SW8240	Volatile Organic Compounds Refer to SW846-8240[a] IRP Handbook[b]	Check of mass spectral ion intensities using BFB	Once per every 12-hour shift	Established criteria in Table 1.10-4 of QAPP	1) Retune instrument. 2) Repeat BFB analysis.
		Five-point calibration (for all analytes)	Initial calibration prior to sample analysis	1) SPCCs[d] average RF ≥0.30[e] (0.25 for bromoform) 2) RSD <30% for CCC[f] RFs	1) Repeat after corrective action if either criterion is not met.
		Daily calibration check	Once per each 12 hours, prior to sample analysis (criteria for these checks must be met prior to sample analysis)	1) SPCCs[d] average RF ≥0.30[e] (0.25 for bromoform) 2) CCC[f] percent difference <25% from average response factors calculated following initial calibration	1) Evaluate system and take corrective action. 2) If source of problem cannot be determined, a new five-point calibration must be generated.
		Internal Standards' (IS) Retention Time (RT) and responses check	All samples, standards, and blanks	RT ±30 seconds at EICP[g] within -50% to + 100% of last calibration verification (12 hrs) for each IS compound	1) Inspect MS or GC for malfunctions. 2) Take appropriate corrective actions. 3) Mandatory reanalysis of samples analyzed while system was malfunctioning unless matrix interferences demonstrated.
		Second source Laboratory Control Sample (LCS) see Section 10.0 QAPP tables for list of analytes spiked	Daily (as per instrument calibration requirements in IRP Handbook[b]). Must be run immediately after each calibration sequence.	Recovery for all analytes within QC Acceptance Criteria in Table 1.10-4	1) Repeat LCS. 2) If still out, stop, identify, and correct problem before proceeding with sample analysis.
		Matrix spike (MS) and matrix spike duplicate (MSD); level of spike should be the larger of the following two criteria: 1) the regulatory concentration limit (if applicable, otherwise 20 µg/L), or 2) the concentration of the daily calibration check.	1 MS and 1 MSD per every 20 Air Force project samples.	QC Acceptance Criteria in Table 1.10-4	1) Check LCS results. 2) If recovery for LCS is outside of designated range, the system is considered to be out of control. Immediate identification and correction of problem is required. 3) Reanalyze any samples affected by out-of-control condition 4) If LCS within criteria, flag data

Table 1.10-12
(Continued)

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action[c]
SW8240 (cont'd)		Surrogate standards spike	Every sample, spike, standard, and reagent blank	Table 1.10-4 criteria	1) Recalculate result 2) If still out, check instrument performance, take corrective action, if necessary. 3) If still out, reanalyze sample 4) Flag result if it does not meet criteria and document in report that steps 1 through 3 were performed.
		Method blank	Daily prior to sample analysis for each matrix and once per extraction batch for medium-level soil sample analysis up to 20 samples/batch	No analytes > method reporting limit except for the following: methylene chloride, toluene, acetone, and MEK which must be ≤5x the MRL.	1) Source of contamination investigated. 2) Appropriate corrective action taken and documented. 3) Repeat initial daily blank analysis and re-extract all medium-level soil samples prior to analysis. 4) Sample results associated with blank contamination are to be flagged.
SW8240 (Cont'd)		MDL study	Once per year	Detection limit established shall not exceed those in Table 2-1 of IRP Handbook[b]	Detection limits that exceed established criteria shall be submitted to the Air Force for approval prior to the analysis of any project samples[b].

[a]Test Methods for Evaluating Solid Waste, U.S. EPA, SW846, September 1986.

[b]IRP Handbook, May 1991.

[c]All corrective actions associated with Air Force project work shall be documented and the records maintained by the laboratory, as specified in the IRP Handbook.

[d]SPCC = System Performance Check Compounds.

[e]SPCC = for bromoform is ≥0.25.

[f]CCC = Calibration Check Compounds.

[g]EICP = Extracted Ion Current Profile.

[h]Methylene chloride, acetone, toluene, and 2-butanone are considered to be common laboratory contaminants. Therefore, corrective action is not required when their presence is detected in laboratory blanks at less then five times the noted reporting limits.

NA = Not applicable.

Table 1.10-13
Summary of Calibration and Internal Quality Control Procedures for SW8260

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action[c]
SW8260	Volatile Organic Compounds Refer to SW846-8260[a] IRP Handbook[b]	Check of mass spectral ion intensities using BFB	Once per every 12-hour shift	Established criteria in Table 1.10-5 of QAPP	1) Retune instrument. 2) Repeat BFB analysis.
		Five-point calibration (for all analytes)	Biannually or when daily calibration fails to meet acceptance criteria	1) SPCCs[d] average RF ≥0.30[e] (0.1 for bromoform, 0.2 for 1,1,2,2-tetrachloroethane) 2) RSD <30% for CCC[f] RFs	1) Repeat after corrective action if either criterion is not met.
		Continuing calibration check standard	Once per each 12 hours	1) SPCCs[d] average RF ≥0.30[e] (0.1 for bromoform, 0.2 for 1,1,2,2-tetrachloroethane) 2) CCC[f] RSD <25%	1) Evaluate system and take corrective action. 2) If source of problem cannot be determined, a new five-point calibration must be generated.
		Internal Standards (IS)	All samples, standards, and blanks	RT ±30 seconds at EICP[g] within -50% to + 100% of last calibration verification (12 hrs) for each IS compound	1) Inspect MS or GC for malfunctions. 2) Take appropriate corrective actions. 3) Mandatory reanalysis of samples analyzed while system was malfunctioning unless matrix interferences demonstrated.
		Second source Laboratory Control Sample (LCS) see Section 10.0 QAPP tables for list of analytes spiked	Daily (as per instrument calibration requirements in IRP Handbook[b]). Must be run immediately after each calibration sequence.	Recovery for all analytes within QC Acceptance Criteria in Table 1.10-5	1) Repeat LCS. 2) If still out, stop, identify, and correct problem before proceeding with sample analysis.

Table 1.10-13
(Continued)

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action[c]
SW82 .0 (Cont'd)		Matri pike (MS) and matrix pike duplicate (MSD); level of spike should be the larger of the following two criteria: 1) the regulatory concentration limit (if applicable, otherwise 20 µg/L), or 2) the concentration of the daily calibration check.	1 MS and 1 MSD per every 20 Air Force project samples.	QC Acceptance Criteria in Table 1.10-5	1) Check LCS results. 2) If recovery for LCS is outside of designated range, the system is considered to be out of control. Immediate identification and correction of problem is required. 3) Reanalyze any samples affected by out-of-control condition 4) If LCS within criteria, flag data
		Surrogate standards pike	Every sample, spike, standard, and method blank	Table 1.10-5 criteria	1) Recalculate result 2) If still out, check instrument performance, take corrective action, if necessary. 3) If still out, reanalyze sample 4) Flag result if it does not meet criteria and document in report that steps 1 through 3 were performed.
		Method blank	Daily prior to sample analysis for each matrix and once per extraction batch for medium-level soil sample analysis up to 20 samples/batch	No analytes > method reporting limit except for the following: methylene chloride, toluene, acetone, and MEK which must be ≤5x the MRL.	1) Source of contamination investigated. 2) Appropriate corrective action taken and documented. 3) Repeat initial daily blank analysis and re-extract all medium-level soil samples prior to analysis. 4) Sample results associated with blank contamination are to be flagged.
		MDL study	Once per year	Detection limit established shall not exceed those in Table 2-1 of IRP Handbook[b]	Detection limits that exceed established criteria shall be submitted to the Air Force for approval prior to the analysis of any project samples[b].

Table 1.10-13
(Continued)

[a]Test Methods for Evaluating Solid Waste, U.S. EPA, SW846, September 1986.

[b]IRP Handbook, May 1991.

[c]All corrective actions associated with Air Force project work shall be documented and the records maintained by the laboratory, as specified in the IRP Handbook.

[d]SPCC = System Performance Check Compounds.

[e]SPCC = for bromoform is ≥0.25.

[f]CCC = Calibration Check Compounds.

[g]EICP = Extracted Ion Current Profile.

[h]Methylene chloride, acetone, toluene, and 2-butanone are considered to be common laboratory contaminants. Therefore, corrective action is not required when their presence is detected in laboratory blanks at less then five times the noted reporting limits.

NA = Not applicable.

Table 1.10-14
Summary of Calibration and Internal Quality Control Procedures for SW8270

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action[c]
SW8270	Semivolatile Organic Compounds Refer to: SW846-3500[a] SW846-3600[a] SW846-8270[a] IRP Handbook[b]	Check of mass spectral ion intensities using DFTPP, (4,4'-DDT, pentachlorophenol and benzidine also to be included in tuning standard to verify injection port inertness and GC column performance)	Initially, prior to calibration, again prior to sample analyses and once per every 12-hour shift	Established criteria in Table 3 of SW846-8270[a] -- Degradation of DDT ≤20% -- Benzidine and pentachlorophenol should be present at their normal responses -- Peak tailing factor ≤3	1) Retune instrument. 2) Clean injection port, if necessary. 3) Remove first 6 to 12 inches of column, if necessary. 4) Repeat analysis tuning standard.
		Five-point calibration for all commercially available analytes	Initial calibration prior to sample analysis	1) %RSD <30% for each individual CCC[e] 2) SPCCs[d] average RF ≥0.050	Repeat after corrective action if any one of the acceptance criteria are not met.
		Daily calibration check	Once per each 12-hour period, prior to sample analysis (criteria for these checks must be met prior to sample analysis). Prepare calibration standards weekly.	1) SPCCs[d] average RF≥0.050 2) CCC[e] percent difference <30%	1) Evaluate system and take corrective action 2) If source of problem cannot be determined, a new five-point calibration must be generated.
		Internal Standards' (IS) Retention Time (RT) and Responses check from calibration check standard.	Immediately after or during data acquisition of calibration check standard.	RT ±30 seconds and EICP[f] within -50% to +100% of last calibration verification (12 hours) for each IS compound	1) Inspect MS or GC for malfunctions 2) Take appropriate corrective actions
		Second source extracted Laboratory Control Sample (LCS) see Section 10.0 tables for list of analytes spiked	Daily (as per Instrument Calibration requirements in IRP Handbook[b]). Must be run immediately after each calibration check.	Recovery for all analytes within QC Acceptance Criteria in Table 1.10-6	1) Repeat LCS 2) If still out, stop, identify, and correct problem before proceeding
		Matrix spike (MS) and matrix spike duplicate (MSD); level of spike should be the larger of the following two criteria: 1) the regulatory concentration limit, if applicable, otherwise near the top of the calibration range, or 2) concentration of ongoing calibration check.	1 MS and 1 MSD per every 20 Air Force project samples	QC Acceptance Criteria in Table 1.10-6	1) Check LCS results. 2) If recovery for LCS is outside of designated range the system is considered to be out of control. Immediate identification and correction of problem is required. 3) Reanalyze any samples affected by out-of-control condition. 4) If LCS within criteria, flag data

Table 1.10-14
(Continued)

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action[c]
SW8270 (cont'd)		Surrogate standards spike.	Every sample, spike, standard, and reagent blank.	Table 1.10-6 criteria. Corrective action required only if more than 1 acid and 1 base surrogate exceeds criteria.	1) Recalculate result. 2) If still out, check instrument performance and take corrective action, if necessary. 3) If still out, reanalyze sample. 4) Flag result and document in report that steps 1 and 3 were performed.
		Method blank	Once per extraction batch (up to 20 sample/batch) and each time there is a change in reagents	No analytes > method reporting limits except phthalates that are <5 times MDL	1) Source of contamination investigated. 2) Appropriate corrective action taken and documented. 3) All samples processed with a contaminated blank are to be reextracted and reanalyzed at no cost to the Air Force. 4) Sample results associated with method blank contamination are to be flagged.
		MDL study	Once per year	Detection limits established shall not exceed those in Table 2-1 of IRP Handbook[b]	Detection limits that exceed established criteria shall be submitted to the Air Force for approval prior to the analysis of any project samples[b]

[a]Test Methods for Evaluating Solid Waste, U.S. EPA, SW846, September 1986.

[b]IRP Handbook, May 1991.

[c]All corrective actions associated with Air Force project work shall be documented and the records maintained by the laboratory, as specified in the IRP Handbook.

[d]SPCC = System Performance Check Compounds.

[e]CCC = Calibration Check Compounds.

[f]EICP = Extracted Ion Current Profile.

NA = Not applicable.

Table 1.10-15
Summary of Calibration and Internal Quality Control Procedures for SW8280

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action[c]
SW8280	Polychlorinated Dioxins and Furans Refer to: SW846-3500[a] SW846-3600[a] SW846-8280[a] IRP Handbook[b]	Mass scale calibration using DFTPP	Initially, prior to calibration, again prior to sample analyses and once per every 12-hour shift	m/e 198, base peak; 199, 5-9% of 198; 275, 10-30 of 198; 365, >1% of 198; 441, present but <443; 442, >40% of mass 198; 443, 17-23% of mass 442	Repeat calibration
		Five-point calibration for all commercially available analytes	Initial calibration prior to sample analysis, and when daily calibration verification fails to meet acceptance criteria.	%RSD ≤15%	Identify and repeat outlying point(s); recalculate curve using repeated points
		Daily calibration check	Once per each 12-hour period, prior to sample analysis	Agreement within 30% of value predicted from multipoint calibration	1) Repeat calibration verification 2) If still out, evaluate system and take corrective action, rerun calibration verification 2) If still out, recalibrate
		Resolution verification with standard containing C_{13}-1,2,3,4-TCDD and C_{13}-2,3,7,8-TCDD	Daily	<25% valley between C_{13}-1,2,3,4-TCDD and C_{13}-2,3,7,8-TCDD	Replace column
		Retention window verification	Daily, unless retention times of internal standards vary <0.2 minutes	<0.2 minute variation of internal standard	Rerun retention time standard, adjust SIM windows
		Sensitivity verification using calibration containing 0.2 µg/mL 1,2,7,8-TCDD	Every 12 hours prior to sample analysis	>50:1 S/N on 320 ion peak from 1,2,7,7-TCDD	Evaluate instrument and take corrective action
		Surrogate standards spike with solution containing C_{13} congeners	Every sample, spike, standard, and method blank	Table 1.10-7 criteria	1) If outside limits, evaluate S/N; if >10:1 and MRLs met and LCS passes, flag 2) If S/N <10:1, run 8% fraction. If 8% fraction surrogates have >10:1 S/N, combine results
		Second source extracted Laboratory Control Sample (LCS) see Section 10.0 tables for list of analytes spiked	Daily (as per Instrument Calibration requirements in IRP Handbook[b]). Must be run immediately after each calibration check.	Recovery for all analytes within QC Acceptance Criteria in Table 1.10-7	1) Repeat LCS 2) If still out, stop, identify, and correct problem before proceeding
		Duplicate Sample	1 oper batch of 20 or fewer samples	50% RPD for surrogates	1) If LCS/LCSD passes, flag data

Table 1.10-15
(Continued)

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action[c]
		Method blank	Once per extraction batch (up to 20 sample/batch) and each time there is a change in reagents	No analytes > method reporting limits	1) Source of contamination investigated. 2) Appropriate corrective action taken and documented. 3) All samples processed with a contaminated blank are to be reextracted and reanalyzed at no cost to the Air Force. 4) Sample results associated with method blank contamination at ≤3 times the detection limit are to be flagged.
		Sensitivity Check	Each sample	>100,000 area count from 334 ion fomr 1 ng injection of C_{13}-1,2,3,4-TCDD	1) Evaluate system 2) Reanalyze sample
		MDL study	Once per year	Detection limits established shall not exceed those in Table 2-1 of IRP Handbook[b]	Detection limits that exceed established criteria shall be submitted to the Air Force for approval prior to the analysis of any project samples[b]

[a]Test Methods for Evaluating Solid Waste, U.S. EPA, SW846, September 1986.

[b]IRP Handbook, May 1991.

[c]All corrective actions associated with Air Force project work shall be documented and the records maintained by the laboratory, as specified in the IRP Handbook.

[d]SPCC = System Performance Check Compounds.

[e]CCC = Calibration Check Compounds.

[f]EICP = Extracted Ion Current Profile.

NA = Not applicable.

Table 1.10-16
Summary of Calibration and Internal Quality Control Procedures for Field Test Kits

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action[c]
Millipore EnviroGard™ PCB Soil Test Kit	PCBs	Positive control(s)	Once every 20 samples	Shades of blue must be lighter than negative control. The higher the concentration of PCB, the lighter the color should be.	Repeat positive and negative controls, and associated samples in affected batch
	Refer to: Manufacturer's Instructions	Negative control	Once every 20 samples	Blue color must develop within 10 minutes after adding substrate	Repeat positive and negative controls, and associated samples in affected batch
		Field duplicates	Every sample	Duplicate test tubes should exibit similar shades of blue	1) If positive and negative controls are acceptable, repeat test for affected sample(s) only 2) If positive or negative controls are not acceptable, repeat test for all samples in affected batch
Millipore EnviroGard™ DDT Soil Test Kit	DDT and metabolites	Positive control(s) (up to 3 points: 0.1, 1, and 10 ppm)	Once every 20 samples	Shades of blue must be lighter than negative control. The higher the concentration of DDT, the lighter the color should be.	Repeat positive and negative controls, and associated samples in affected batch
	Refer to: Manufacturer's Instructions	Negative control	Once every 20 samples	Blue color must develop within 10 minutes after adding substrate	Repeat positive and negative controls, and associated samples in affected batch
		Field duplicates	Every sample	Duplicate test tubes should exibit similar shades of blue	1) If positive and negative controls are acceptable, repeat test for affected sample(s) only 2) If positive or negative controls are not acceptable, repeat test for all samples in affected batch

[a]Test Methods for Evaluating Solid Waste, U.S. EPA, SW846, September 1986.

[b]IRP Handbook, May 1991.

[c]All corrective actions associated with Air Force project work shall be documented and the records maintained by the laboratory, as specified in the IRP Handbook.

NA = Not Applicable.

Corrective Action Report (CAR)

Part I Initial Information (Furnished by Originator)

Originator: _____ Urgency Level:

SAM #: _____ Method #: _____ Requires resolution for immediate job ☐

Client: _____ Matrix: _____ Requires resolution for future jobs ☐

Date:

To Person Responsible for Action: _____

Present Situation Requiring Action:

Site / Lab: Type: QC Limit ☐ Documentation ☐ System ☐ Other ☐

Date / Time Identified:

Description of Situation: (attach supporting data if available)

Recommended Corrective Action or Improvement:

Description: Implemented by:

Technical Director Copy (White)

Figure 1.14-1. Corrective Action Report

Part II Reply / Resolution (Furnished by Technical Director)

Proposed by: Date: CAR #:

Description: Scheduled Implementation:

Part III Implemented Correction Action (by:)

Description: Date Implemented:

Part IV Follow-Up Required: Yes ☐ No ☐ (by:)

Verified by: Date: Comments:

Information Copies, Distributed by Technical Director:

Figure 1.14-1. (Continued)

RESEARCH & ENGINEERING

RECOMMENDATION FOR CORRECTIVE ACTION

A. Initial Information

RCA NO.:	DATE:	URGENCY LEVEL ☐
ORIGINATOR:	APPROVED BY:	1. Potential for major data loss or invalidation. 2. Potential for failure to achieve data quality objectives. 3. Suggested improvement.
ORGANIZATION/INDIVIDUAL RESPONSIBLE FOR ACTION:		

B. Problem Identification

SITE/LAB:	SYSTEM:	DATE PROBLEM IDENTIFIED:
DESCRIPTION OF PROBLEM:		

C. Recommended Corrective Action

DESCRIPTION:	IMPLEMENT BY:

D. Problem Resolution

PLANNED CORRECTIVE ACTION:	PROPOSED BY:	DATE PROPOSED:	SCHEDULED IMPLEMENTATION:
IMPLEMENTED CORRECTIVE ACTION:			DATE IMPLEMENTED:

E. QA Verification

VERIFIED BY:	DATE:	COMMENTS:

White: Return for Implemented Corrective Action Yellow: Return for Planned Corrective Action Pink: File copy Gold: Originator's Copy

Information Copies Distribution _____

Figure 1.14-2. Recommendation for Corrective Action

- Significant QA problems and recommended action.

Two types of QC results will be reported as appropriate for each sampling and analytical task:

- Sampling QC
  - — Field blank analyses
  - — Trip blank analyses
  - — Ambient blank analyses
  - — Field duplicate sample analyses
- Analytical QC
  - — Method spike analyses
  - — Laboratory duplicate analyses
  - — Matrix spike analyses
  - — Matrix spike duplicate analyses
  - — Reagent blank analyses
  - — Laboratory check sample analyses
  - — Surrogate spike analyses

Tables summarizing pertinent QC data for the task will be prepared. The range of the results for each type of data (blanks, spikes), the total number of samples, and number of acceptable results will be indicated.

An evaluation of project data with regards to QC results also will be provided in the technical report. This evaluation will present guidelines for data usability during site interpretation and risk assessment in terms of bias and imprecision.

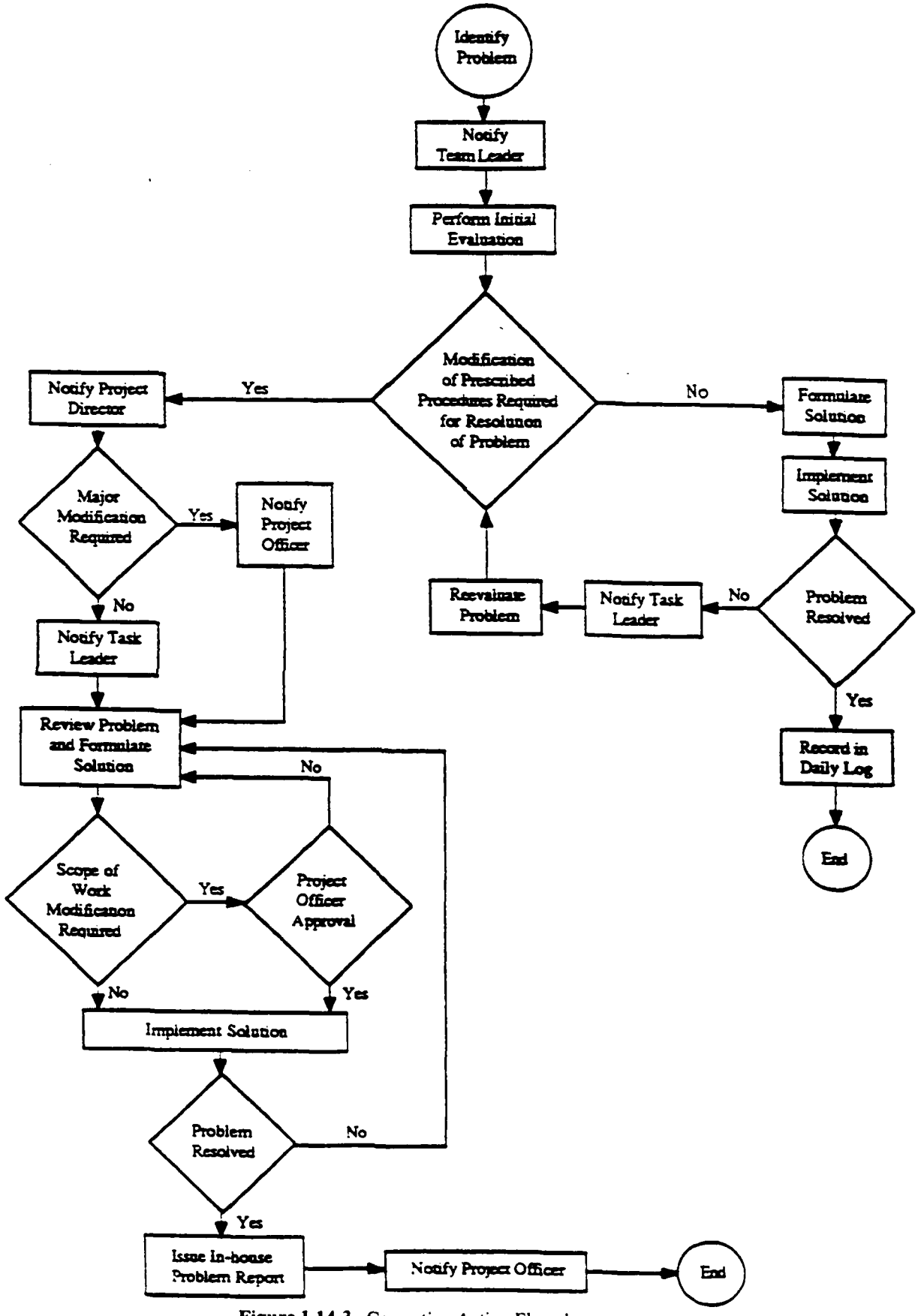

Figure 1.14-3. Corrective Action Flowchart

Section 2
FIELD SAMPLING PLAN

This section of the 1994 Addendum to the Sampling and Analysis Plan (SAP) provides requirements and procedures for all of the field activities to be conducted during the 1994 field season. This document is intended to augment the original Field Sampling Plan (FSP), which was included as section 2.0 to the *Installation Restoration Program Stage 3 SAP for Galena and Campion Air Force Stations* (Radian, June 1992).

The primary sampling goals of the 1994 field season include groundwater sampling at Galena Airport and surface and shallow subsurface soil sampling at Galena Airport and Kalakaket Creek RRS. No drilling activities, such as monitoring well installation or soil borings, will be conducted. Tables 2-1 and 2-2 summarize the proposed soil and water sampling activities, respectively, for Galena Airport and Kalakaket RRS. Figure 2-1 shows the relative location of the two installations. The 1994 Addendum to the Work Plan, under separate cover, provides maps and descriptions of all new investigations areas. The *Installation Restoration Program Work Plan, Galena and Campion Air Force Stations, Alaska* (Radian, June 1992) and the *Remedial Investigation/Feasibility Study Draft Technical Memorandum, Galena Airport and Campion Air Force Station, Alaska* (Radian, May 1994) provide maps and descriptions of all previously investigated sites.

2.1 Site Documentation

The first field activity to be performed at each site or sampling location is to note the field conditions. For Galena Airport locations, this entails describing changes to the location since previous sampling events. For Kalakaket Creek RRS, detailed descriptions of all field observations must be recorded. The following should be noted, as applicable:

- Vegetation, including any evidence of stress;
- Surface topography, including erosion, drainage ways, and standing water; and
- Any debris or staining on the ground surface, or sheen on standing water.

Photographs will be taken to document these observations whenever possible.

2.2 Soil Sampling

Surface soil sampling will be conducted at several locations at both Galena Airport and Kalakaket Creek RRS. Samples will be screened using several different methods and sent to the laboratory for confirmation. Based on screening or laboratory results, deeper samples (up to 5 ft) may be collected using a hand auger.

2.2.1 Soil Screening

Four types of soil screening will be used to help direct the soil sampling efforts. The following paragraphs describe the screening methods and their application. Analytical methods for the field test kits are included in Appendix A.

Headspace Analysis/Organic Vapor Screening

Field screening of samples by headspace analysis may be performed using an HNu® photoionization detector (PID) or equivalent organic vapor monitor (OVM). Before use each day, the OVM will be calibrated in the field following the manufacturers' detailed calibration instructions (see Section 1.7 of the QAPP).

To perform the headspace analysis:

1) Place sample in a glass mason jar, filling it 50-60% full.

2) Seal the jar using aluminum foil and the outer ring of the lid.

3) Allow the sample to equilibrate for up to one hour and reach ambient temperature.

Table 2-1
Summary of Soil Sampling Activities at Kalakaket Creek RRS and Galena Airport, Alaska 1994

Sampling Locations	Field Screenings			Laboratory Analysis						
	TPH	PCB	DDT/Pest.	AK101	AK102	8240	8270	8080	6010	8280
KALAKAKET CREEK										
Ambient Location									4	
Antenna Day Tanks	8			2	2					
Vehicle Maintenance Garage	8			4	4	4	4		4	
Paint Storage Building						4	4	4	4	
Equipment Building	10	10		4	4	4		4	4	
Equipment Building Transformers		10						4		
Diesel Tank Fill Area	6			3	3					
Septic Tank Outfall				1	1	1	1	1	1	
Drum Storage Area	18	18	18	4	4	4	4	4	4	
Temporary Garage	8			2	2				2	
Temporary Diesel Tank	4			1	1					
Water Pump House	4			1	1					
SUBTOTALS	66	38	18	22	22	17	13	17	23	0
GALENA										
Fire Protection Training Area										6
Main Base			30					15		
Pump Station Outfall				4	4	4	4	4	4	
DDT Stockpiled Soil								15		
SUBTOTALS	0	0	30	4	4	4	4	34	4	6
TOTALS	**66**	**38**	**48**	**26**	**26**	**21**	**17**	**51**	**27**	**6**
Galena	**GROUNDWATER SAMPLING**									
Monitoring Wells				38	38	38[a]	32	38	6[b]	
Base Water Supply Wells				3	3	3[a]	3	3	0	
TOTALS	**0**	**0**	**0**	**41**	**41**	**41[a]**	**35**	**41**	**6[b]**	**0**

Table 2-2
Summary of Water Sampling Activities at Galena Airport, Alaska 1994

Sampling Locations	AK101	AK102	8260	8270	8080	6010/7060/7421
Ambient Location						2
FPTA	6	6	6		6	
POL Storage Area	10	10	10	10	10	
West Unit	19	19	19	19	19	
Control Tower Drum Storage Area	3	3	3	3	3	3
Base Water Supply Wells	3	3	3	3	3	
TOTALS	41	41	41	35	41	6

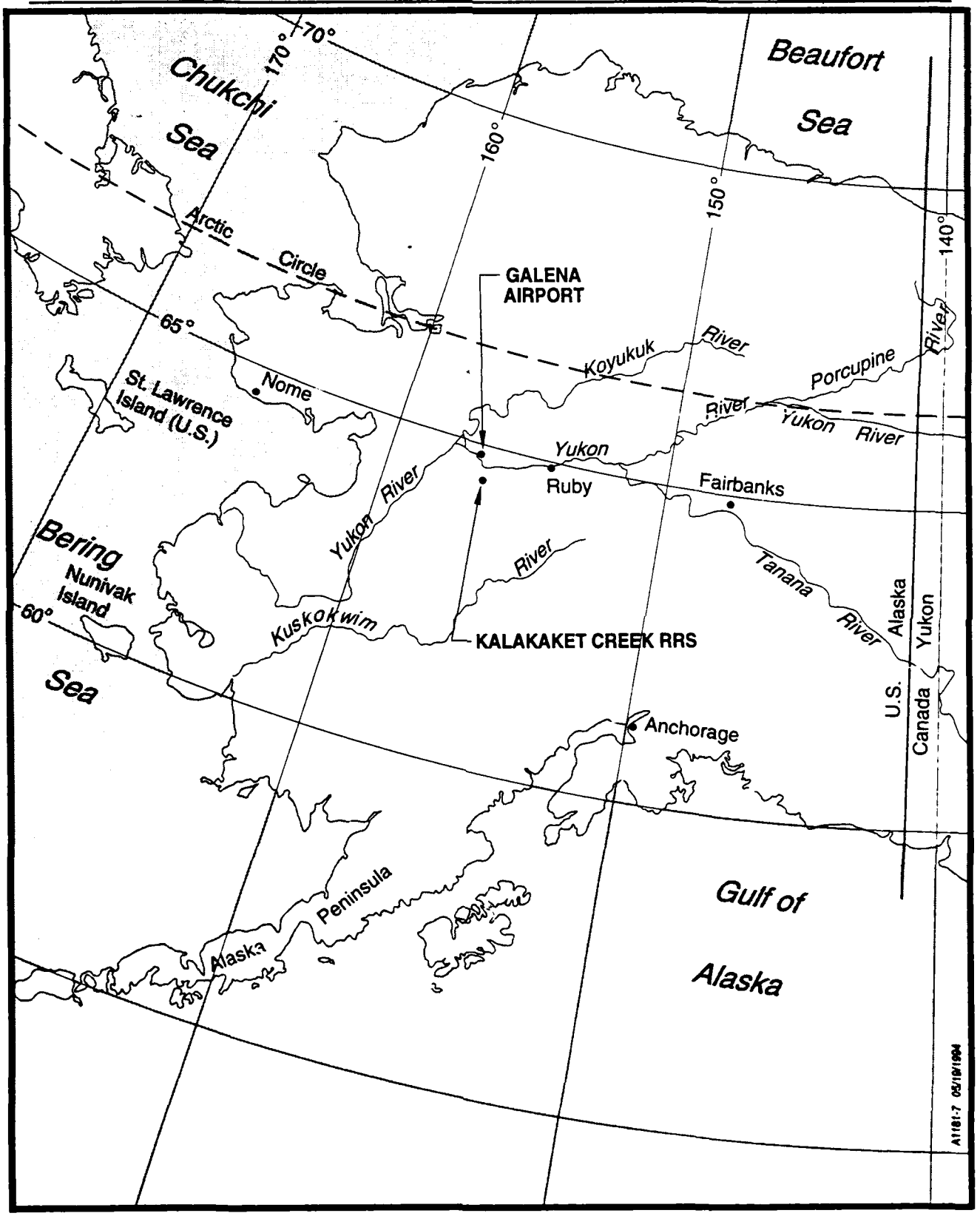

Figure 2-1. Location of Galena Airport and Kalakaket Creek RRS

4) Pierce the aluminum with the OVM probe, using caution not to insert the probe into the soil.

5) Record the measurement from the gauge or digital display into the field logbook.

This method will allow for the very quick and economical screening of soil samples for volatile organic compounds (VOCs).

Total Petroleum Hydrocarbon (TPH) Screening

An infrared (IR) method for determining TPH will be used to screen soil samples from several locations at the Kalakaket Creek RRS. The analyses will be performed using an General Analysis Corporation (GAC) IR analyzer, and the results will be used to determine which samples will be submitted for laboratory confirmation. For each sampling location where TPH is detected in screening samples, at least one confirmation sample will be sent to the laboratory to determine the accuracy of this method. Appendix A includes a complete copy of this screening method.

Polychlorinated Biphenyl (PCB) Screening

Soil samples will be collected from three locations at the Kalakaket Creek RRS for PCB screening. A total of 38 soil samples from the equipment building transformer area, and the three drum storage areas (DSAs) will be analyzed using a PCB immunoassay field test kit. Based on the results of the field screening, approximately 12 samples will be submitted for laboratory confirmation. A copy of the PCB immunoassay method is provided in Appendix A.

Total DDT Screening

Field screening for total DDT (DDT + DDD + DDE) will be conducted at 30 locations within the Galena Airport main base triangle and 6 locations at each of 3 DSAs at Kalakaket Creek RRS. The DDT screening locations within the main base triangle will be based on previous sampling locations and will be placed to identify DDT "hotspots" of approximately 5000 sq ft (approximately 70 ft by 70 ft). Total DDT will be determined in the field using an immunoassay field test kit that detects total DDT at 0.1, 1, and 10 ppm. A copy of the method is provided in Appendix A.

2.2.2 Surface and Shallow Subsurface Soil Sampling

A stainless steel hand trowel will be used to collect surface soil samples from within 6 in. of the ground surface. Hand augers equipped with 3-in. diameter cylindrical stainless steel bits will be used to collect soil samples from depths of up to 5 ft below ground level (bgl). Samples for VOC analysis will be collected as soon as the sample is retrieved from the auger or trowel. Soil samples will be collected as follows:

1. Check all equipment and sample containers to ensure that the equipment is clean and that the containers are new and have been properly prepared.

2. Label all sample containers.

3. Scrape away all surface debris from an area of approximately 1 sq ft.

4. Dig, or auger, to the desired sampling depth.

5. For discrete samples:
   a. At the desired sampling depth, collect soil directly from the the bit or trowel for all VOC analyses.
   b. Place the remainder of the soil sample into a stainless steel bowl and composite. If necessary, use a properly decontaminated no. 4 sieve to remove excess gravel and cobbles. Fill remaining sample jars with composited sample.

6. Note in a field notebook the interval from which the sample was collected and describe the soil (i.e., silty sand, sandy gravel, etc.).

7. Decontaminate the trowel or auger using the following procedure:
   a. Wash in a solution of potable water and Alconox, or equivalent laboratory grade detergent. Scrub with brushes.
   b. Rinse with copious amounts of potable water.
   c. Rinse with reagent grade deionized water.
   d. Rinse with pesticide grade methanol, followed by pesticide grade hexane.
   e. Allow the equipment to air dry and store in aluminum foil if not used immediately.

2.3 Groundwater Sampling

Groundwater samples will be collected from 40 monitoring wells and 3 base water supply wells. Groundwater sampling procedures include field instrument calibration, water-level and free-product measurements, well purging, and sample collection. Activities required to sample the monitoring wells will be completed by a field team of two people. This will expedite the sample collection process and promote safety in the field.

2.3.1 Sample Container and Trip Blank Preparation

Prior to leaving for the field, trip blank samples will be prepared by the laboratory as required. These trip blanks will be carried to the well head along with other sampling supplies, and should accompany the samples from the time of collection to analysis. The purpose and preparation of trip blank samples are discussed in section 2.4.1.

Sample containers will be prepared with preservatives prior to leaving for the sampling location. Coolers filled with an adequate amount of ice should also be prepared pior to leaving for the well head.

2.3.2 Water Level and Free Product Measurements

Prior to sampling a monitoring well, the static water level measurement should be determined. The following procedure should be followed:

- Upon arrival at the well head, note any damage to the security posts or casing, and whether the lock is damaged or missing.
- Unlock the well cover.
- Uncap the well and measure the organic vapor content of the casing air and breathing zone with an OVM and upgrade protective equipment if necessary as described in the *Health and Safety Plan, Galena and Campion Air Force Stations, Alaska* (Radian, July 1993).
- If the OVM reading exceeds 100 ppm in the casing, use a properly decontaminated water-product interface meter to determine if free product is present. If there is free product in the well, measure the thickness and record it on the sampling log. Sound the bottom of the well using the interface probe.
- If the OVM reading is less than 100 ppm, use a decontaminated water-level indicator tape to measure the depth to water; repeat the water-level measurement until two consecutive measurements agree within 0.01 ft. Sound the bottom of the well using the water level probe and record all measurements on the sampling log.

2.3.3 Well Purging

Each monitoring well will be purged immediately prior to sample collection. This ensures the sample consists of fresh formation water rather than stagnant water that has been stored in the well casing. During all sampling activities, well purging equipment will be positioned so that any potential volatile organic sources, such as vehicles, are downwind of the well. This minimizes contamination caused by entrainment of volatile contaminants in the sample. Any potential sources of volatile organics that are unavoidable will be noted on the Groundwater Quality Sampling Log (Figure 2-2), and an ambient blank will be collected at that sampling location. The purpose of and procedure for collecting ambient blanks are described in section 2.4.1.

GROUNDWATER QUALITY SAMPLING LOG

Project: **Galena Airport RI/FS**
Client: **AFCEE**

Well ID: _____ Location: _____
Date: _____ Weather _____
Time: _____ Samplers: _____

Comments: _____

Field Measurements:

OVM Reading (ppm): _____ Product Depth (ft btoc): _____
Water Depth (ft btoc): _____ Well Depth (ft btoc): _____
Product Thickness (ft): _____ Saturated Thickness (ft): _____
Borehole Volume (gal): _____ 3 Borehole Volumes (gal): _____

Purge/Sample Method: _____

Time	Cum. Vol. (gal)	Temp. (deg C)	pH (pH units)	Conductivity (umhos/cm)	Comments:

Final Measurements:

Temperature _____ pH _____ Conductivity _____ Alkalinity _____

Figure 2-2. Groundwater Quality Sampling Log

If no dedicated sampling equipment is present in the well, clean, unused Waterra® tubing and foot valve will be installed. A properly decontaminated Teflon® bailer may be used to purge wells with very short water columns. A minimum of three wetted borehole volumes of water will be purged from each well. A wetted borehole volume, in gallons, is calculated using the following formula:

$$V = [(3.14b^2L - 3.14c^2L)(0.30) + (3.14c^2L)] * 7.48 \text{ gal./ft}^3$$

where:

V = One wetted borehole volume (gal.);
b = Borehole radius (ft);
c = Casing radius (ft);
L = Height of water column in well (ft);

and the porosity of the filter pack is assumed to be 30 percent. For 2-in. diameter wells with 8-in. borehole, one wetted borehole volume equals approximately 0.9 gal./ft of water column. Using this calculation, a well with 20 ft of wetted casing would have one wetted borehole volume of 18 gal., requiring a total purge volume of 54 gal.

To determine when stabilization has occurred, pH, temperature, and conductivity will be monitored on a regular basis during purging until two successive readings of all three parameters do not vary by more than ±0.1 pH unit, ±1.0°C, and ±5% change in micromhos/cm, respectively. If stabilization does not occur, samples may be collected after a total of six wellbore volumes have been removed from the well. To promote consistency in the field data, one person will measure and record all water parameter data while the other purges the well.

In low-yield wells which are purged dry before three well bore volumes have been removed, the sample will be collected as soon as a sufficient amount of water has re-entered the well to collect samples. The time at which the well is purged dry will be recorded on the groundwater field sheets, as well as the volume of water removed prior to sampling.

A calibrated 5-gal. bucket (or similar container of known capacity) will be used to measure the amount of water being removed from the well during the purging process. Elapsed time will be noted as the container is filled, thereby allowing the calculation of the discharge rate. The total amount of water purged from each well will be recorded on the Groundwater Quality Sampling Logs (see Figure 2-2). All purge water will be put in 55-gal drums which are sequentially numbered and clearly labeled with the date, contents, well ID, OVM reading, and volume. The drum number, date of generation, and contents will be promptly recorded in the Investigation Derived Wastes (IDW) drum log.

2.3.4 Groundwater Field Measurements

Final pH, temperature, and conductivity measurements will be taken and recorded prior to sampling the well. A colorimetric alkalinity titration will also be performed at the well head. The pH and conductivity probes, thermometer, and beaker or other container will be rinsed with deionized water and the sample to be tested prior to making the measurements. Values for pH, conductivity, and temperature will be recorded with a minimum accuracy of ±0.1 pH unit, 5% in micromhos/cm, and ±1.0°C, respectively.

2.3.5 Groundwater Sample Collection

One of two types of sampling equipment, a dedicated Waterra inertial pump system or a bailer, will be used to collect groundwater samples from the monitoring wells. Dedicated sampling systems will be used for individual wells, minimizing the potential of cross-contamination to other wells. Wells equipped with Waterra pumps will be sampled directly from the outflow. Wells with a very short water column may be sampled using a decontaminated Teflon bailer. A stainless-steel or Teflon-coated leader (8 ft length) will be securely attached to the bailer if a bailer is used. The bailer will be lowered slowly into the well, taking care to cause as little disturbance as possible to the water surface. Water will be collected near the middle of the water column. As the bailer is lowered and raised, the sampler will be careful to keep the line clean and off the ground surface. To minimize this problem, the polypropylene line (tied to the Teflon leader) can be directed into a clean bucket or similar

container as the bailer is being raised in the well. A bottom emptying device may be used to transfer water from the bailer into the sample containers.

Groundwat~ nples will be recovered in a prearranged priority, so that all collection and handling takes place as efficiently as possible. Prior to collecting a sample from the discharge line or bailer, the samplers will wear new, clean protective gloves to avoid cross contamination. Care will be taken to minimize disturbance of the groundwater. Samples will be taken in the following order to minimize the loss of volatile compounds:

- Volatile organic compounds (AK101 and 8260);
- Semivolatile compounds (AK102 and 8270); and
- Pesticides and PCBs (8080).

The sample bottles for VOCs must be filled slowly to prevent the entrapment of air bubbles, splashing, or agitation of the water. Care will be taken to avoid touching the mouth of the discharge line, the top of the sample bottle, the inside of the cap, or the Teflon septa. A septum that falls out of the cap onto the ground cannot be used. The bottle will be filled completely such that a meniscus forms. The cap will be screwed on and the bottle inverted, tapped firmly, and checked for the presence of air bubbles. Accurate analytical results for volatile organic compounds may be compromised if there is any free air trapped in the sample container.

Before leaving the well head, the sampling team will ensure that the well is secured with a keyed-alike padlock, the well ID is clearly discernible, and all sampling-related debris is removed from the area. Also, the drum containing the purge water will be clearly labeled, on both the lid and sides.

2.3.6 Groundwater Sampling Equipme.. Decontamination Procedures

Dedicated sampling equipment will be used to the fullest extent possible to avoid cross contamination of monitoring wells and samples. For nondedicated equipment, such as bailers and electronic water level meters, proper decontamination procedures will be followed. Equipment will be washed in a potable water and Alconox (or equivalent laboratory grade detergent) solution and rinsed with copious amounts of potable water. This is followed by an ASTM Type II reagent grade water rinse. The equipment is then rinsed with pesticide grade methanol, followed by pesticide grade hexane, and allowed to air dry. All pH and conductivity meter probes, thermometers, and measurement containers will be thoroughly rinsed with ASTM Type II reagent water and the new sample water before taking additional measurements. Clean, disposable gloves will be worn during and after decontamination to avoid contamination of equipment.

2.4 Field QA/QC

Field QA/QC will be controlled by compliance to sample holding times, sample preservation requirements, and by periodic field QC samples. Table 1.5-1 in the QAPP (Section 1) details the holding times and preservation requirements for each of the analytical methods. The following subsections describe the QC samples that will be collected and other field QA/QC measures.

2.4.1 QC Samples

Five types of field QC samples will be collected during the investigation: trip blanks, ambient blanks, equipment blanks, field duplicates, and matrix spike/matrix spike duplicates (MS/MSDs). The number, type, and composition of these samples will comply with the requirements outlined in the QAPP (Section 1.0 of this document). The following paragraphs describe the five types of QC samples.

Trip Blanks

A trip blank is a VOC sample bottle filled in the laboratory with Type II reagent grade water, transported to the site, handled like a sample and returned to the laboratory for analysis. Trip blanks will not be opened in the field. The trip blank for soils is the same as for water samples. One trip blank will accompany every cooler of soil and water samples sent to the laboratory for the analysis of VOCs. Coolers will be arranged to minimize the number of

trip blanks needed. These blanks will be analyzed for VOCs only.

Ambient Blanks

An ambient blank is Type II reagent grade water that is poured into a sample container at a sampling site. It will be handled like a sample and transported to a laboratory for analysis. Ambient blanks will be collected when samples are collected downwind of possible VOC sources such as active runways or roadways. Ambient blanks need not be taken at every site. These blanks will be analyzed for VOCs only.

Equipment Blanks

An equipment blank is Type II reagent grade water that is poured into or pumped through a decontaminated sampling device, transferred to a sample bottle, and transported to a laboratory for analysis. Equipment blanks will be taken to address the effectiveness of sampling equipment decontamination, and need only be taken when nondedicated sampling equipment is used. These blanks will be analyzed for all laboratory analyses requested for environmental samples collected at the site.

Field Duplicates

Field duplicates are two samples collected independently at a sampling location during a single act of sampling. Ten percent of all groundwater samples will be field duplicates to determine matrix and analytical variability. As directed by AFCEE, no field duplicates of soil samples will be collected. Field duplicates will be identified so that laboratory personnel are unable to distinguish them from normal field samples. Both members of the duplicate pair will be analyzed for the same parameters in the laboratory. In addition, field parameters (pH, conductivity, temperature, and alkalinity) will be determined for the duplicate samples to determine the reproduceability of the field data.

MS/MSDs

MS/MSDs are sample aliquots that are provided to the laboratory to assess matrix interferences. An aliquot of the sample is spiked by the analyst with known concentrations of all analytes of interest. The spiking occurs prior to sample preparation and analysis. The recovery of the spike is calculated to determine the bias of a method in a given sample matrix.

2.4.2 Field Measurement Instrument Calibration

Conductivity meters, pH meters, thermometers, and OVMs will be used during groundwater sampling. The conductivity meters and pH meters will be calibrated on a daily basis at the beginning of each field day. The conductivity meter will be calibrated to a standard solution according to the manufacturers instructions. The pH meter will calibrated and adjusted with at least two buffer solutions which bracket the expected sample pH. A single point calibration check using the pH 7 buffer is performed at each well prior to collecting a final measurement. If the meter drift is 0.2 pH units or greater, the two point calibration is repeated. Any instrument drift at the end of the day is also noted in the calibration field log. Calibration procedures for all field equipment are detailed in Section 1.7 of the QAPP.

2.5 Sample Labeling, Chain of Custody, Storage, and Transportation

The following paragraphs describe the procedures required for proper labeling, documentation, and shipping of viable samples.

2.5.1 Labeling

Labels will be computer generated, completed in the field using a waterproof permanent marker, and securely attached to the sample jar. All samples will be clearly labeled with the following information:

- Project name/client;
- Sample location;
- Sample type (analytical method);
- Preservatives used;
- Sampler's initials; and
- Date and time of collection.

Adhesive tape will be used, if necessary, to secure labels. In no case will tape be used to seal sample containers.

2.5.2 Chain of Custody

Field personnel will maintain chain-of-custody records for all field and field QC samples. A sample is defined as being under a person's custody if any of the following conditions exist:

- It is in their physical possession;
- It is in their view, after being in their physical possession;
- It was in their possession, and they locked it up or otherwise sealed it so that tampering would be evident; or
- It is in a designated secure area.

Field personnel will complete a chain-of-custody record for each sample. The chain-of-custody form will accompany each sample shipment container from the field to the laboratory to establish the documentation needed to trace sample possession. Figure 2-3 shows an example of the chain-of-custody form which will be used for the Galena Airport and Kalakaket Creek RRS.

Upon arrival at the designated laboratory, the chain-of-custody form will be completed with:

- Name of the person receiving the container and date of arrival or receipt of samples;
- Name of the person opening the shipping container, along with date, time, temperature, and condition of shipping container; and
- Any remarks regarding sample condition upon arrival.

All sample coolers will be sealed in a manner that will prevent and detect tampering.

2.5.3 Sample Storage and Transportation

Samples will be packaged, shipped, and stored in a manner which avoids contamination and ensures sample integrity. All samples are stored in coolers on ice or in properly monitored refrigerators from immediately after collection until analysis. A properly monitored refrigerator contains a National Institute of Standards and Testing (NIST)-traceable thermometer that is checked daily to assure that sample temperature is maintained at or below 4°C. The refrigerator must be in a secure area and access limited to the field sampling team.

When packaging samples for commercial transport, an absorbent material such as vermiculite will be used to minimize the effect of any breakage and to absorb any spills. Protective packaging will be used on sample bottles to minimize the risk of breakage during transport, and to ensure that the samples do not freeze. Sample packaging requirements for hazardous materials requiring interstate transport are defined in the *Code of Federal Regulations* (CFR) 49, Chapter 1, Part 171 and will be utilized, if required, during sample transport. The samples from this field effort are not expected to be classified as hazardous.

2.6 Field Activity Staking and Surveying

The surveying of locations for all field activities will be measured by a certified land surveyor as the distance in feet from a reference location that is tied to the state plane coordinate system, whenever possible. The initial surveying task will consist of establishing a benchmark and baseline, and locating and staking (marking) the location of each predetermined soil sampling location. Some locations may be initially surveyed using a Global Positioning System (GPS) capable of establishing coordinates to an accuracy within 2 ft. In particular, a GPS may be used to establish control at Kalakaket Creek RRS. Also, the location and elevation of each monitoring well installed during previous investigations will be verified and resurveyed as necessary. Whenever possible, all survey locations will be numbered and marked using a stake. If staking is not possible, survey locations will be marked with an alternate method.

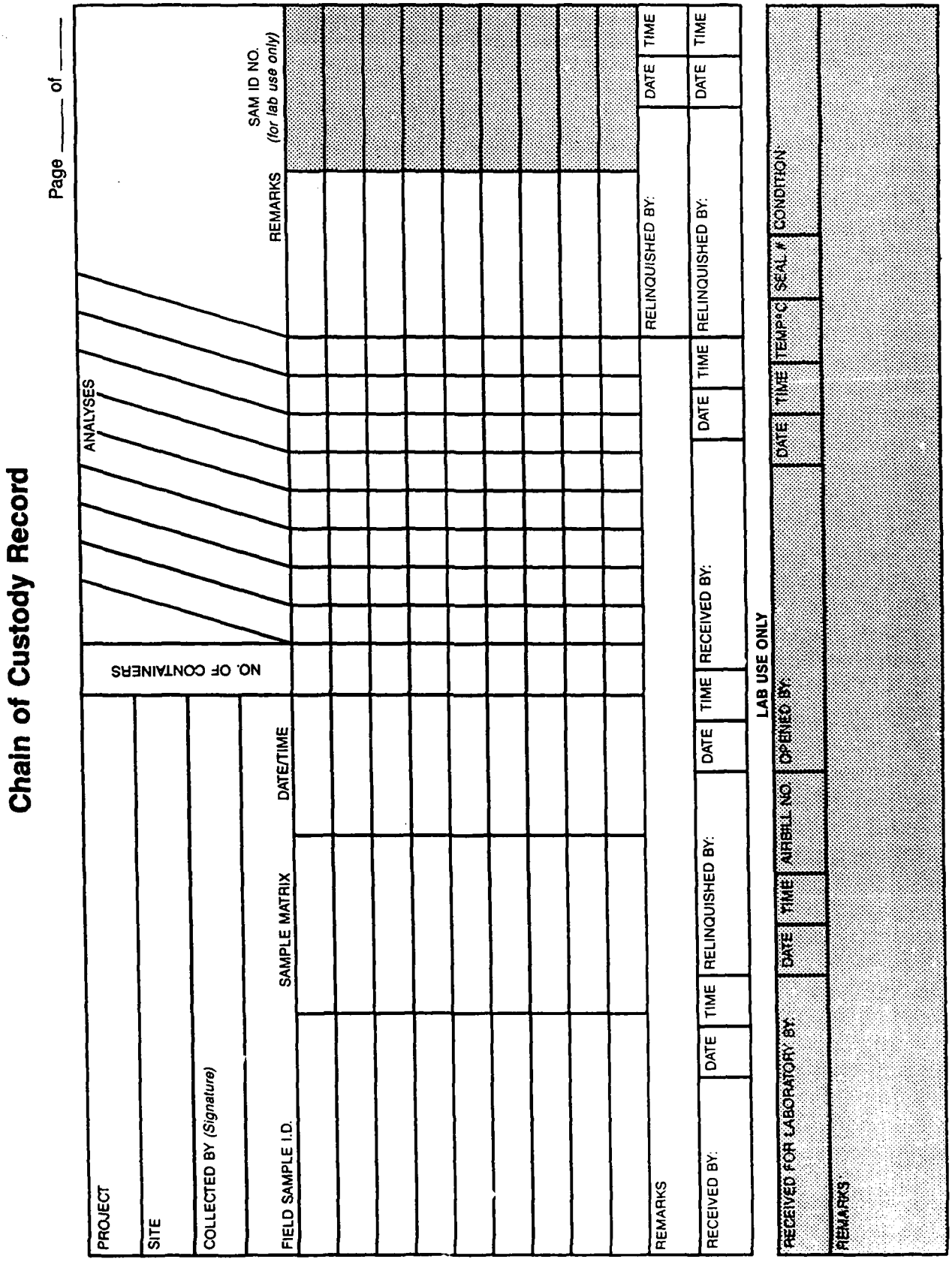

Figure 2-3. Chain-of-Custody Form

2.7 Site Management

This section briefly discusses the general aspects of Site Management including record keeping, management of IDW, the identification of key Air Force personnel, and the duties of the field sampling team.

2.7.1 Recordkeeping

Field records shall be maintained which will enable the recreation of all sampling and measurement activities performed during the RI. The field investigation sampling and analysis activities will be designed to meet the requirements of the Installation Restoration Program Information Management System (IRPIMS). In addition, specific data requirements for certain activities as listed in the following sections will be met. All information will be recorded with indelible ink in a permanently bound, numbered notebook with sequentially numbered pages. These records will be archived during and upon completion of the project in an easily accessible form and will be available, upon request, to the Air Force.

The records kept for all activities conducted during the RI will include the location, date and time, identity of people performing the activity, and weather conditions. For all field measurements, the numerical value and units of each measurement, the type of instrument, and the calibration results will be recorded. Notes on all sampling activity will include: sample type and sampling method used, sample identity and depth(s) of collection, sample amount, a sample description (grain size, color, odor, clarity, etc.), identification of sampling device, and any uncontrollable conditions which may affect the sample integrity (weather, air quality, well damage, etc.).

2.7.2 Containerization, Storage, and Treatment or Disposal of IDW

IDW will be properly placed in 55-gal. drums and clearly labeled and numbered at the point of generation. Prior to the end of the field effort, these wastes will be moved to a staging area designated by the Air Force. The Air Force has the responsibility for treatment or disposal of the IDW, and will follow appropriate guidelines similar to those put forth in the memorandum dated 24 September 1993 (Radina, 1993).

2.7.3 Air Force Personnel and Support

Base IRP Project Manager (RPM)
Mr. Wes Lannen
611th CES
21885 2nd Street
Elmendorf AFB, AK 99506
(907) 552-4532

Alaska Restoration Team Chief
Contracting Officer's Representative
Mr. Samer Karmi
AFCEE\ERDW
8001 Inner Circle, Suite 2
Brooks AFB, TX 78235-5328
(210) 536-5297

The base will provide the following support:

1. Assign an accumulation point within the installation to which the contractor can deliver containers holding any drill cuttings or well installation/development/decontamination fluids generated from the required work which are suspected to be hazardous.

2. Treat or dispose of any IDW.

3. Provide a pre-construction briefing to the field team members to acquaint them with local rules/policies/procedures, and introduce local points of contact.

4. Arrange for the following:

   a. Access to existing engineering plans, drawings, diagrams, aerial photographs, digitized map files, etc., to facilitate evaluation of IRP sites under investigation.
   b. A staging area for storing supplies.
   c. A temporary office, laboratory, and storage facility with access to electricity, phone lines, and a potable water source.

2.7.4 Coordination of Field Activities

Field tasks will require the organization, scheduling, and other field logistics for contractor personnel and subcontractors. An experienced geologist or engineer will provide program logistics and communication between the Air Force, the field teams, subcontractors, state inspection personnel, and task management regarding the routine, daily activities. In addition, the duties of the field sampling team members will be defined to allow for efficient completion of the work:

Supervising Geologist

- Supervises sampling activities;
- Act as on-site health and safety officer;
- Supervises or conducts screening of soil samples;
- Inspects samples for physical evidence of contamination;
- Makes decisions regarding sample selection for analyses based on lithology, field screening measurements, and visual inspection;
- Keeps daily log of operations in field notebook, including complete daily field reports, time and materials log, and soil sample and OVA and/or OVM reading data sheet;
- Describes soil samples;
- Records other pertinent information on sampling log form (e.g., OVM readings, discoloration, odor, and waste observed);
- Records intervals from which samples were taken for analysis;
- Prepares field notes and well logs for entry into geologic database; and
- Assists in making field decisions regarding location of sampling, etc.

Field Assistant

As assistant to the Supervising Geologist, the Field Assistant:

- Places sample labels on sample containers that will be submitted for analyses;
- Assists in performing soil head-space analyses;
- Assists in well purging and sampling;
- Assists in packaging of samples for laboratory shipment;
- Performs health and safety monitoring measurements in the immediate work zone using an OVM;
- Completes health and safety data in field logbook and on appropriate field forms; and
- Decontaminates sampling tools (trowels, sieves, etc.).

2.7.5 Clearance and Permits

It is not anticipated that any site clearance or permits will be required, as none of the field activities require drilling or other intrusive techniques.

2.8 Site Health and Safety

A detailed discussion of site health and safety for Kalakaket Creek RRS is presented under separate cover as the Kalakaket Creek PA/SI Health and Safety Plan. Health and safety issues for Galena Airport are covered in the *Health and Safety Plan, Galena and Campion Air Force Stations, Alaska* (Radian, July 1993).

Section 3
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Appendix

ANALYTICAL METHODS

METHOD FOR THE DETERMINATION OF GASOLINE RANGE ORGANICS

1. Scope and Application

1.1 Analytes

1.1.1 This method is designed to measure the concentration of gasoline range organics (GRO) in water and soil. This corresponds to an alkane range from the peak start of C_6 to the peak start of C_{10} and a boiling point range between approximately 60°C and 170°C.

1.1.2 Components greater than or equal to C_{10} present in products such as diesel or fuel oil are detectable under the conditions of the method. If, based on a review of the chromatogram, the presence of these product types is suspected, the client should be informed. Gasoline or other specific products or compounds may be identified by the use of pattern recognition techniques. This may include, but is not limited to, analysis of additional reference materials. These additional efforts are not contained within this method.

1.1.3 With the optional photo ionization detector (PID), this method can be extended for specific determination of volatile aromatics (BTEX) as specified in EPA methods 602 and 8020.

1.2 Quantitation Limits

1.2.1 The Practical Quantitation Limit (PQL) of this method for gasoline range organics is approximately 5 mg/Kg GRO as gasoline for soils and 0.1 mg/L GRO as gasoline for water.

1.3 Dynamic Range

1.3.1 Dilutions should be performed as necessary to put the chromatographic envelope within the linear range of the method. In general, the approximate range is 0.5 to 2 mg/L of gasoline.

1.4 Experience

1.4.1 This method is based on a purge-and-trap, Gas Chromatography (GC) procedure. This method should be used by, or under supervision of, analysts experienced in the use of purge-and-trap systems and gas chromatographs. The analysts should be skilled

in the interpretation of gas chromatograms and their use as a quantitative tool.

2. Summary of Method

2.1 This method provides gas chromatographic conditions for the detection of volatile petroleum fractions such as gasoline. Samples are analyzed utilizing purge-and-trap sample concentration. The gas chromatograph is temperature programmed to facilitate separation of organic compounds. Detection is achieved by a flame ionization detector (FID) or PID/FID in series. Quantitation is performed by comparing the total chromatographic area between and including C_6 (hexane) and C_9 (nonane), through the peak start time of C_{10}, including resolved and unresolved components, based on FID response to a blended commercial gasoline standard.

2.2 Water samples can be analyzed directly for gasoline range organics by purge-and-trap extraction and gas chromatography. Soil or waste samples are dispersed in methanol to dissolve the volatile organic constituents. A portion of the methanol solution is analyzed as in the water method.

2.3 Special field sampling techniques are required to minimize the loss of volatiles from soil resulting from conventional sampling and sample handling techniques.

2.4 This method is based in part on U.S. EPA SW-846 [1] methods 5030, 8000, 8020, 8015, a single laboratory method evaluation study conducted by the American Petroleum Institute [2], work by the EPA Total Petroleum Hydrocarbons Methods Committee [3], and work by the State of Alaska, Department of Environmental Conservation, Juneau Environmental Analysis Laboratory with support from the Underground Storage Tank Program.

3. Definitions

3.1 Gasoline Range Organics (GRO): All chromatographic peaks, both resolved and unresolved, eluting between the peak start time for C_6 (hexane) and the peak start time for C_{10} (decane). Quantitation is based on a direct comparison of the baseline - baseline integrated area within this range to the total area of the calibration standard using FID response.

3.2 Gasoline Calibration Standard (GCS): An equal-weight mixture of unleaded, leaded, and premium commercial gasolines mixed and diluted to appropriate concentrations, used to prepare a standard curve. In those

areas where leaded gasoline is not longer available, a second unleaded regular gasoline may be used to prepare the calibration standard.

3.3 Laboratory Control Standard (LCS): A commercially prepared, certified gasoline quality control standard (ERA Certified, or equivalent) used by the laboratory as a quality control check to verify the accuracy of the external calibration. Alternatively, a NIST traceable, multi-component synthetic organic standard (API - PS 6) may be used (see component Table 3 in appendix).

3.4 Surrogate Control Standard (SCS): Either bromofluorobenzene or trifluorotoluene, or a mixture of both, used as a laboratory control and to normalize GRO concentrations.

3.5 Surrogate Control Sample: A reagent water or method blank sample spiked with the SCS used in the method. The surrogate recovery is used to evaluate method control (see 7.3).

3.6 Laboratory Control Sample: A reagent water or method blank sample spiked with a commercial gasoline other than the ones blended to prepare the GCS. The spike recovery is used to evaluate method control. The LCA may be used in the Laboratory Control Sample.

3.7 Pattern Recognition Standards: Various commercial gasolines and other petroleum products used by the laboratory to identify petroleum products.

3.8 Normal Alkane Standard (NAS): A normal alkane standard (C_6 through C_{10}) which is analyzed once per 12 hour analytical "day" with each set (analytical batch) of samples, not to exceed 10 samples per set. This standard is used to verify expected boiling point ranges for petroleum products, establishes the retention time window for quantitation of GRO and provides data for column performance. The compounds of BTEX and NAS can be combined if desired, and if all quality control criteria are met (see section 10).

3.9 Other terms are as defined in SW-846 [1].

4. Interferences

4.1 High levels of heavier petroleum products such as diesel or heating fuel may contain some volatile components producing a response within the retention time range for GRO. Other organic compounds, including chlorinated solvents, ketones, and ethers are also detectable by this

method. As defined in the method, the GRO results include these compounds.

4.2 Samples contaminated with a single compound which is detectable between C_6 and C_{10} (e.g., some solvents) will be quantitated against gasoline standard, resulting in a value which may be biased high for that compound. If the compound can be identified, a gross quantitation based on response factor may be performed using a standard of the identified compound. This is a suggestion only and not a requirement of the method (see 9.9.4).

4.3 Samples can become contaminated by diffusion of volatile organics during shipment and storage. A trip blank prepared from reagent water (for water samples) or methanol with Ottawa sand (for soil and sediment samples) and carried through sampling and subsequent storage and handling can serve as a check for such contamination.

4.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe and/or purging device must be rinsed between samples with reagent water and methanol. Whenever an unusually concentrated sample is encountered, it should be followed by analysis of a solvent blank or reagent water to check for contamination. For volatile samples containing high concentrations of water-soluble materials, suspended solids, high boiling compounds or organohalides, it may be necessary to wash the syringe or purging device with a detergent solution, rinse with distilled water and methanol, and then dry in a 105°C oven between analyses. The trap and other parts of the system are also subject to contamination. Therefore, frequent bake-out and purge of the entire system may be required. A screening of all samples prior to analysis is recommended to protect analytical instrumentation (see 9.6.1).

5. Safety Issues

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets should also be made available to all personnel involved in chemical analyses. Additional references to laboratory safety should be made available and identified for the information of the analyst.

6. Apparatus and Materials

6.1 Glassware (All specifications are suggested only.)

6.1.1 40 mL glass vials with Teflon lined septa and screw caps (aka, VOA or VOC vials).

6.1.2 4 oz amber glass wide mouth jars with Teflon lined septa which are fused to the screw caps.

6.1.3 Volumetric flasks, class A: 10 mL, 50 mL, 100 mL, 500 mL and 1000 mL with ground glass stoppers.

6.1.4 Disposable pipettes: Pasteur.

6.2 Syringes

6.2.1 5 mL Leurlock glass syringe and 5 mL gas-tight syringe with shutoff valve.

6.2.2 For purging large sample volumes for low detection limit analysis, 25 or 50 mL syringes may be used. Remember to adjust other volumes as necessary throughout the method.

6.2.3 Microsyringes: 1, 5, 10, 25, 100, 250, 500 and 1000 uL.

6.3 Analytical balance, capable of accurately weighing to the nearest 0.0001 g for preparation of standards, and a top-loading balance capable of weighing to the nearest 0.1 g for samples.

6.4 Stainless steel spatula.

6.5 Gas Chromatography

6.5.1 Gas Chromatograph: Analytical system complete with gas chromatograph suitable for purge-and-trap sample introduction and all required accessories, including detectors (FID required, additional PID optional), column supplies, gases and syringes. A data system capable of determining peak areas using a forced baseline and baseline projection is required. A data system capable of storing and reintegrating chromatographic data is recommended.

6.5.2 Columns:

6.5.2.1 Column 1: 105-m x 0.53 mm ID. Restek RTX 502.2, 0.3 micron film thickness or equivalent.

6.5.2.2 Capillary columns are required to achieve necessary resolution. The column must resolve 2-methylpentane from the methanol solvent front in a midrange LCS standard and must resolve ethylbenzene from m/p-xylene. Some columns may require sub ambient cooling to achieve these guidelines.

6.5.2.3 Column performance will be evaluated by separation number. The separation number should be greater than 20 for octane and nonane. Inadequate column performance can distort chromatograms, cause errors in quantitation and qualitative results, and produce distorted surrogate peaks.

$$\text{Separation Number} = \frac{RT_{oct} - RT_{non}}{W_{½,\,oct} + W_{½,\,non}} - 1$$

Where:

$W_{½}$ = peak width at half height

RT = retention time

6.5.2.4 The column must be capable of separating typical gasoline components from the surrogate and (optional) internal standard.

6.5.3 Purge-and trap device: The purge-and-trap device consists of three separate items: the sample purger (sparging device), the trap, and the desorber (furnace). Several complete assemblies are commercially available. See Table 1 for summary.

6.5.3.1 **Purging chamber**: The recommended purging chamber is designed to accept 5 mL samples with a water column at least 3-cm deep. The gaseous headspace between the water column and the trap must have a total volume of less than or equal to 15 mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3-mm at the origin. The purge gas must be introduced no more than 5-mm from the base of the water column.

6.5.3.2 **Trap**: The trap must be at least 25-cm long and have an inside diameter of at least 0.105 in. Starting from the inlet, the trap should be packed with the following adsorbents: 1/3 2,6-diphenylene oxide polymer (60/80 mesh, chromato-graphic grade, Tenax GC or equivalent), 1/3 silica gel (35/60 mesh, Davison grade 15 or equivalent), and 1/3 coconut charcoal (Barnebey Cheney, CA-580-26 lot #M-2649 or equivalent, prepared by crushing through 26 mesh screen). It is recommended that 1.0 cm of methyl silicone-coated packing (OV-1 (3%) on chromosorb-W, 60/80 mesh or equivalent) be inserted at the inlet to extend the life of the trap. Prior to initial use, the trap should be conditioned overnight at 180°C by back flushing with an inert gas flow of at least 20 mL/min. Vent the trap effluent to the hood, not to the analytical column. Prior to daily use, the trap should be conditioned for 10 min. at 180°C with back flushing. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples.

An alternate trap uses 7.6-cm Carbopack B and 1.3-cm Carbosieve S-III (Supelco Cat# 2-0321R). This trap should be desorbed at 240°C and baked to 300°C.

6.5.3.3 **Desorber (Furnace)**: The desorber should be capable of rapidly heating the trap to 180°C for desorption. The polymer section of the trap should not be heated higher than 180°C, and the remaining sections should not exceed 220°C during bake-out mode.

6.5.4 The purge-and-trap device may be assembled as a separate unit or may be coupled to a gas chromatograph, as long as complete transfer of the sample is assured.

7. Reagents and Standards

7.1 Reagent Water: Carbon-filtered, purged water which has been shown to be free from purgable compounds (this has also been called organic-free water). Nitrogen or helium may serve as purge gas.

7.2 Methanol: Pesticide grade or equivalent. Store away from other solvents.

7.3 Stock Standard Solutions - Prepare the following stock standards. Unless noted, all are prepared using the methanol listed in 7.2 as solvent. Standard preparation should follow guidelines in SW 846 [1]. All standards must be stored without headspace at -10 to -20°C and protected from light. Properly stored standards must be replaced within 6 months of preparation. Standards should be checked regularly to assure their integrity.

7.3.1 Internal Standard: An internal standard (1-chloro-4-fluorobenzene) is recommended for 602/8020 quantitation on the PID. Due to potential interferences, the internal standard is not recommended for GRO (FID) quantitation.

7.3.2 Recommended Surrogate Standard: 50 ug/mL of bromofluorobenzene and/or trifluorotoluene (SCS). Add 5.0 uL of this surrogate directly into the 5 mL syringe with every water sample and reference standard analyzed. Surrogate is spiked into soil samples during the extraction step (see 8.2.1).

7.3.3 Normal Alkane Standard: At a minimum, this multi-component blend of alkanes serves as a retention time window defining mix for GRO. The concentration of the individual components should not be less than 500 ug/mL and not more than 1000 ug/mL. Additional standards may be added to this mix if 602 or 8020 is to be done concomitantly.

7.3.4 Gasoline Calibration Standard: A mixture of equal weights of leaded, unleaded and supreme gasolines from different refiners serves as the calibration standard. No fewer than 3 concentrations of this GCS are diluted directly into the 5 mL syringe (linear range approx. 0.5 to 2.0 mg/L) at the time of calibration. Other than one standard concentration near the method detection limit, the expected range of concentrations found in real samples should define the working range of the GC (see 9.3.2).

7.3.5 Stock Standard for Laboratory Control Samples: From a commercial gasoline other than those used to prepare the GCS, prepare a dilution of 500 ug/mL in methanol. Addition of the following amounts yields the indicated concentrations:

0.005 mL added to 5 mL water:	0.5 mg/L
0.5 mL added to 10 g soil:	25 mg/Kg

8. Sample Collection, Preservation, Handling and Holding Times

8.1 Aqueous Samples:

8.1.1 Aqueous samples should be collected in triplicate without agitation and without headspace in contaminant-free, amber glass 40-mL vials with Teflon-lined septa in the caps. If amber glass vials are not available, clear glass may be substituted if the samples are protected from light. The Teflon layer must contact the sample (zero headspace). Sample vials should contain 200 uL of 50% HCl as a preservation for volatile analytes. Refrigerated samples (4° ± 2° C) must be analyzed within 14 days after collection.

8.1.2 A travel blank (contaminant-free amber glass 40-mL vial with Teflon-lined septum, filled to zero headspace with purged, organic free water) should accompany all sampling kits at a minimum ratio of 1 for every 10 samples collected and should be analyzed with the field samples.

8.2 Soils and Sediments: Soil and sediment samples require special procedures to minimize the loss of volatiles during transit from the field to laboratory.

8.2.1 Soil or sediment samples should be collected with minimum disturbance into tared 4 oz (or larger, if appropriate) jars with a Teflon lined septum fused to the lid. 25 mL aliquots of methanol (includes 1.2 mL of a surrogate solution at 50 ug/mL) should be carefully added to the undisturbed soil until the sample is submerged. It is extremely important that the weight of the jar, the weight of the methanol/surrogate solution and the weight of the sample collected be known. Absorbent, organic soils such as muskeg and tundra will require a higher methanol-to-sample ratio, while beach sand may tolerate a lower ratio. Soil for volatiles analysis can be collected using any coring device which minimizes soil disturbance. Any scraping, stirring or similar activity will result in a loss of volatiles during sampling. A sufficient number of samples should be collected to provide for backup in the event of breakage. Although it is not necessary to refrigerate all samples at 4°C after collection and until analysis is complete, collected samples must be kept below 25 °C. A second surrogate added to the methanol and soil mixture after sample collection may be used in addition to, but not in place of, the surrogate with which the field methanol was prepared.

8.2.2 A reagent methanol travel blank should be prepared in the same manner as the sample vials, with the addition of 10 g of Ottawa sand or other appropriate soil or sediment (Norwood loam, Houston black clay, or other standard soil). Every effort must be made to match the matrix of the travel blank to the anticipated matrix of the field samples for validity and comparability. Travel blanks should accompany all sampling kits at a ratio of 1 for every 10 samples collected.

8.2.3 Field blanks may be added to the sampling protocol, and prepared by the laboratory in the same manner as travel blanks are prepared, as required by the Assessment Firm or the Project Manager.

8.2.4 A sample of the same soil as will be analyzed for GRO should be collected into a wide mouth glass jar with a moisture proof lid for moisture determination and oversized gravel materials determination (if appropriate). This sample should be processed at the same time as the volatiles sample is analyzed.

8.2.5 Travel blanks, field blanks, method blanks, etc. should be prepared from the same batch of solvent, reagents and vials as are used for sample collection.

8.3 An API study [2] has indicated that soil samples collected into methanol with zero headspace can be held for up to 28 days at 4°C with no apparent losses. This has been verified by others [4]. Therefore, 28 days is the maximum holding time for soil/sediment samples collected into methanol.

8.4 Because the jars and methanol are pre-weighed to determine the quantity of soil collected for analysis, it is extremely important that the sampler put evidence tape on the kit ONLY and not on the individual bottles. Removal of evidence tape is extremely difficult and the additional weight biases final results.

8.5 Travel blanks, field blanks and bottle blanks should be prepared as appropriate to meet the quality assurance goals of the project plan.

9. Procedure

9.1 Volatile compounds are introduced into the gas chromatograph by purge-and-trap. Purge gas (nitrogen or helium) should be set at a flow rate of 25 - 40 mL/min. Purge time is set at 12 min.

9.2 Waters:

9.2.1 Purge-and-trap may be used directly on most water samples.

9.2.2 Water samples high in dispersed sediments (non-settling or slow settling solids) must NOT be filtered before analysis, as this results in loss of volatiles. In most cases, a muddy water sample can be left undisturbed until the solids settle out. Then an aliquot of the sample can be taken with a 5 mL gas tight syringe, being careful to not disturb the sediment layer. Introduction of sediment into the purge device can result in occlusion of the frit, leading to incomplete purging of the sample and low-biased results. In any case, sample preparation should be noted, and an approximate volume given for the solids, if present.

9.3 Soils and Sediments:

9.3.1 Soils and solids are methanol extracted. An aliquot of the extract is added to reagent water, and analyzed as in 9.10.

9.3.2 Samples should be collected into tared, methanol and surrogate containing sample jars (see 8.2).

9.3.3 The entire volume of soil should be submerged in methanol/surrogate solution.

9.3.4 Weigh the sample jar upon receipt and record the total filled weight. Swirl the jar gently for 2 minutes to be sure that the soil sample is dispersed into the methanol, and allow the sediment to settle. It is recommended that the meniscus of the methanol be marked and dated on the outside of the jar.

9.3.5 Best results are obtained by allowing the sample volatiles to equilibrate with the methanol for at least 48 hours before continuing with the analysis. However, this is not always possible. In any case, note the time difference between when the methanol was delivered into the soil sample and when analysis was initiated.

9.4 Improperly Collected Soils and Sediments:

9.4.1 When solids are collected by the sampling techniques in SW-846 [1], volatile results are biased low. Therefore, data from these samples (collected without methanol preservative) must be reported as "greater than or equal to" the calculated mg/Kg GRO as gasoline, and may not be accepted as valid by State project managers.

9.4.2 In order to prepare extracts from these types of collection containers, gently mix the contents of the sample container with a narrow metal spatula. Do not discard any supernatant liquids, as the entire contents of the sample container must be represented.

9.4.3 For sediment/soil and waste that are insoluble in methanol, weigh 10 g (wet weight) of sample into a tared 20 mL vial, using a top loading balance. Note and record the actual weight to 0.1 g.

9.4.4 Quickly add 9.5 mL of methanol and 0.5 mL of the 50 ug/mL surrogate spiking solution to the vial (or, after adding spiking solution, fill to the line on the volumetric flask), cap and swirl (do not shake) for 2 min.

9.4.5 Allow sediment to settle. Note alternate sample preparation procedure on data transmittal.

Note: These steps must be performed rapidly and without interruption, in a laboratory free from solvent fumes, to avoid loss of volatile organics or cross contamination.

9.5 Methanol Soluble Solids:

9.5.1 For waste that is soluble in methanol weigh 1 g (wet weight), to the nearest 0.1 g, into a tared 10 mL volumetric flask.

9.5.2 Quickly add 9.5 mL of methanol and 0.5 mL of the 50 ug/mL surrogate spiking solution to the vial (or, after adding spiking solution, fill to the line on the volumetric flask), cap and swirl for 2 minutes, to disburse the waste into the methanol.

9.5.3 Allow sediment to settle, pipette an aliquot to an amber glass vial for storage at 4°C (zero headspace).

9.6 Sample Screening:

9.6.1 It is highly recommended that all samples be screened prior to analysis, as these samples may contain enough petroleum product to overload the column and/or detector(s). This screening step may be AK100, analysis of a solid sample's methanol extract (diluted) using AK101, the headspace method (SW-846 Method 3810 [1]) or the hexadecane extraction and screening method (SW-846 Method 3820 [1]).

9.7 Gas Chromatography conditions (recommended)

9.7.1 Column 1: Set helium column pressure to 20#. Set column temperature to 30°C for 1 min., then ramp at a rate of 5°C/min. to 100°C, then 8°C/min. to 240°C and hold for 7.5 min. Conditions may be altered to improve the resolution of gasoline range organics.

9.7.2 Other columns: Set GC conditions to meet the criteria in 6.5.2.2.

9.8 Calibration:

9.8.1 The GC system should be set up as in Section 9.2 This should be performed prior to calibration or to final preparation of the samples or sample extracts for analysis.

9.8.2 The calibration curve must be represented by no less than 3 concentrations of Gasoline Calibration Standard (a 5 point calibration curve is recommended). Prepare final solutions of GCS and SCS directly in a 5-mL glass syringe containing reagent water in the following manner: Using a microsyringe, add the aliquot of calibration standard directly to the reagent water in the glass syringe (refer to 9.10.7) by inserting the needle through the syringe opening. When discharging the contents of the microsyringe, be sure that the tip of the needle is well beneath the surface of the reagent water in order to prevent escape of calibration standard components. Similarly, add 5.0-uL of the 50 ug/mL Surrogate Control Standard. Inject the prepared dilution(s) into the purge vessel(s) through the two way valve, and proceed with calibration.

9.8.3 Choose GCS concentrations to cover the GRO range expected in the samples or the linear range of the instrument, whichever is lesser. One of the concentrations must be near the method detection limit. Due to potential carry over, do not purge more than 10 ug of gasoline in 5 mL of water (2 mg/L). A calibration concentration at 0.01 mg/L (0.5 to 1.5 ug/L for individual volatiles) is recommended for additional quantitation if 602/8020 is to be included.

9.8.4 Tabulate the area response of the gasoline against mass injected. The ratio of the response to the amount injected, defined as the response factor (RF), can be calculated for the standard at each concentration. If the percent relative standard deviation (%RSD) is less than 25% over the working range, linearity through the origin can be assumed, and the continuing calibration response factor

can be used in place of a calibration curve. Use the average response factor from the calibration curve as reference.

$$\text{Response Factor} = \frac{\text{Total area of gasoline standard}}{\text{Gasoline standard amount (mg/mL)}}$$

9.8.5 The calibration curve must be confirmed using the Laboratory Control Standard. This certified (or traceable) standard verifies the accuracy of the calibration. The concentration of the LCS should be within the expected concentration range of the samples to be analyzed.

9.8.6 The working calibration curve or response factor must be verified on each working day or every 12 hours (whichever is more frequent) by the injection of a midpoint continuing calibration standard (CCS). If the response factor for the method standard varies from the response from the calibration curve by more than 25% a new calibration curve must be prepared.

$$\text{Percent Difference} = \frac{R_1 - R_2}{R_{avg}} \times 100$$

where:

R_1 = Average RF from the calibration curve.

R_2 = Response factor from CCS.

$$R_{avg.} = \frac{(R_1 + R_2)}{2}$$

9.9 Retention Time Window and Pattern Recognition

9.9.1 Before establishing windows, be certain that the GC system is within optimum operating conditions (6.5). Make three injections of the gasoline alkane standard (7.3.3) throughout the course of a 72 hour period. Serial injections over less than a 72 hour period result in retention time windows that are too tight.

9.9.2 Calculate the standard deviation of the three absolute retention times for each NAS component and for the surrogate.

9.9.2.1 The retention time window for individual peaks is defined as plus or minus three times the standard deviation of the absolute retention time for each component.

9.9.2.2 In those cases where the standard deviation for a particular analyte is zero, the laboratory should use ±0.05 min. as a retention time window.

9.9.3 The laboratory must calculate retention time windows for each standard on each GC column and whenever a new GC column is installed or instrument conditions changed. The data must be retained by the laboratory and updated regularly (no less frequently than once a year).

9.9.4 Pattern Recognition: The experience of the analyst weighs heavily in the interpretation of the chromatogram. References 6, 7 and 8 contain some information on hydrocarbon pattern recognition. Environmental samples may contain more than one type of product, and loss of light end components may indicate that the product has been in the subsurface a longer period of time. It may also make product identification using GRO tentative at best. In these cases, and if product identification is essential to the goals of the project, a GC/MS Oil Identification method utilizing the technique of selected ion extraction is recommended. GC/MS Oil Identification is beyond the scope of this method.

9.9.4.1 The analyst must generate a value for both gasoline range organics and gasoline, if possible, for each sample analyzed. Identification of gasoline is performed by comparing the retention times and patterns of the peaks in the sample chromatogram to those in the standard product chromatogram. This information must be reported on the final data transmittal. At a minimum, a boiling point range and the boiling point of the area of maximum response must be noted.

9.9.4.2 Quantitation of the gasoline range organics is based on summed, baseline-baseline integration of all resolved and unresolved peaks eluting between the peak start of C_6 and the peak start of C_{10}. Qualitative identification of gasoline may include all the same peaks or the analyst may eliminate some peaks. For instance, peaks could be deleted due to unusual peak shape. Other analyses, such as GC/MS, may be used to identify interferences.

9.9.4.3 Note: Although the retention time window definition introduces a low bias (55 to 75% for gasoline in Ottawa Sand), it improves precision and reduces interferences from heavier petroleum components.

9.10 Gas Chromatograph Analysis:

9.10.1 Samples are analyzed by GC/FID. Water, with or without methanol extract, to be analyzed for GRO is introduced into the programmed gas chromatograph (section 9.2) using purge-and-trap sample concentration.

9.10.2 If initial calibration (9.8) has been performed, verify the calibration by analysis of a mid-point CCS. With each day's run, open a 12 hour analysis window. This is done by running the NAS.

9.10.3 A gasoline LCS at a concentration representative of the field samples being analyzed must also be run once every 10 samples and at the end of each sequence. If the result does not fall within 25% of the true value, all samples run before the failed QC must be reanalyzed.

9.10.4 Calculate the percent difference of the response factor from the mean response factor for each analyte to be quantitated (as in 9.8.4). This is done for gasoline as a "group" from the CCS if GRO only is to be quantitated, and for each of the 10 components in the NAS standard if additional quantitation is required. If the response factors have a difference greater than 25%, the instrument must be recalibrated (9.3).

9.10.5 A reagent water blank must be run in every sequence to determine the area generated from normal baseline noise under the conditions prevailing within the 12 hour period. Add 100 uL of methanol to the blank when soil or sediment extracts are to be analyzed. The noise area is generated by projecting a horizontal baseline between the retention times observed between the beginning of hexane and the beginning of decane. This lab control sample is integrated over the GRO area in the same manner as for the field samples, and is reported as the instrument or reagent blank. Do not blank subtract. This information is for data interpretation purposes only.

9.10.6 Blanks should also be run after samples suspected of being highly concentrated, in order to prevent carryover. If the blank analysis shows contamination above original baseline levels, the trap and column must be baked out and subsequent blanks analyzed until

the system is shown to be free from contaminants.

9.10.7 Water samples may be introduced into the system in the following manner:

9.10.7.1 Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample or standard bottle, which has been allowed to come to ambient temperature, and pour the sample into the syringe using caution to not agitate the sample which would result in loss of volatiles. Replace the plunger and compress the sample. Invert the syringe so that the air bubble rises to the top (valve end) of the syringe. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. Add 5 uL surrogate spiking solution through the valve bore of the syringe and proceed with analysis.

9.10.7.2 This process of taking an aliquot destroys the validity of the liquid sample for future analysis. Therefore, if there is only one 40-mL vial of sample, the analyst should fill a second syringe at the same time the first one is prepared, in the same manner, to protect against possible loss of sample integrity. This second sample is maintained at 4°C with valve closed only until such time as the analyst has determined that the first sample has been analyzed successfully. If a second analysis is needed, it must be from the second syringe and must be analyzed within 24 hours of the opening of the original sample vial. Care must be taken to prevent air from leaking into (and to prevent volatiles from leaking out of) the syringe containing the backup aliquot.

9.10.8 Methanol extracts from soils or sediments must be diluted into reagent water for analysis, as are methanol soluble dilutions. A table is provided at the end of the method to help determine the volume of methanol extract to add to the 5 mL volume of regent water, in order to keep the response of the major constituents in the upper half of the linear range of the curve. (See Table 2) The maximum volume of methanol extract usable per 5 mL purge volume is 100 uL (this is used in calculating the PQL, section 1.2).

9.10.8.1 Follow directions for filling a syringe as outlined in 9.10.7.1, except use reagent water instead of sample. Introduce desired volume of methanol extract by inserting the needle of a microsyringe through the valve

opening of the reagent water filled 5 mL syringe, and depressing the micropipette plunger when the needle is well below the surface of the reagent water. The surrogate has already been added (see 8.2). Proceed with analysis.

9.10.9 Dilutions:

9.10.9.1 If the product concentration exceeds the linear range of the method as defined by the range of the calibration curve, the sample (or extract or dilution) must be diluted and reanalyzed. The response of the major peaks should be kept in the upper half of the linear range of the calibration curve.

9.10.9.2 It is most desirable to adjust the volume of extract introduced into the reagent water as in 9.10.8.1 in order to compensate for concentrated sample extracts. However, if that is not possible, the following procedure is appropriate for diluting samples. All steps must be performed without delays until the diluted sample is in a gas-tight syringe:

9.10.9.3 Dilutions may be made in class A volumetric flasks (10 mL to 100 mL seem most useful). Select the volumetric flask that will allow for the necessary dilution. Although intermediate dilutions may be necessary for highly concentrated samples, remember that the more transfers the sample makes, the greater the chance components will be lost.

9.10.9.4 Calculate the approximate volume of reagent water to be added to the volumetric flask selected and add slightly less than this volume of reagent water to the flask.

9.10.9.5 Inject the proper aliquot of sample from the syringe prepared in Paragraph 9.10.7.2 into the flask. Aliquots of less than 1-mL are not recommended for dilution of water samples using this method. Make sure aliquot is introduced well below the surface of the reagent water in the volumetric flask to minimize sample loss.

9.10.9.6 Dilute the sample to the mark with reagent water, disturbing the surface as little as possible. Cap the flask and invert three times. Repeat the above procedure for additional dilutions. Continue as in 9.10.7.

9.10.10 Alternative Dilution Technique:

9.10.10.1 Alternatively, the dilutions can be made directly in the glass syringe to avoid loss of volatiles. If diluting methanol extracts, follow 9.10.8 using a smaller volume of extract in the 5 mL purge volume or the procedure outlined for the dilution of water samples.

9.10.10.2 Attach a syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject sample into the purging chamber. Proceed with the analysis. For additional information, refer to purge-and-trap methods in SW-846 [1].

9.11 Moisture Determination for *Solids*

9.11.1 If oversized gravel materials (greater than 1/4" in diameter) are present, use AK201.

9.11.2 Moisture determinations should accompany all soils data (reported in mg/dry Kg) so the client can, at will, determine the results in the original soil condition. Reporting in mg/dry Kg can best be done if an unpreserved portion of the sample (collected without methanol) has been provided. Because of the potential for high gasoline or related compound concentrations in the soil, all drying should be done under a functioning hood.

9.11.3 In order to determine % moisture, pre-weigh an aluminum weighing boat. Weigh 5-10 g of the sample into the boat and record both weights to the nearest 0.001 g. Dry the sample overnight in a warm (105°C) oven.

9.11.4 Remove the sample from the oven and cool in a desiccator until the sample reaches room temperature and weigh to the nearest 0.001g. Record the weight.

9.11.5 Return the soil sample to the oven for an additional time period (not less than 2 hours), cool again in the desiccator until the sample reaches room temperature and weigh to the nearest 0.001g.

9.11.6 If the weight of the sample has remained constant (± 4%) from the initial "dry" weight (9.11.4), use this number for the moisture determination (see 9.12.2). If the second weighing shows that the sample has lost further weight, continue drying and weighing the sample until the weight becomes constant, then proceed to 9.12.2.

9.12 Calculations:

9.12.1 External Standard Calibration:

The concentration of Gasoline Range Organics in the sample is determined by calculating the absolute weight of analyte purged, from a summation of peak response for all chromatographic peaks, resolved and unresolved, eluting between the peak start time for C_6 (hexane) and the peak start time for C_{10} (decane), using the calibration curve or the calibration factor determined in 9.8 and baseline-baseline projection. Refer to Section 9.9 (Retention Time Window and Pattern Recognition). The concentration of Gasoline Range Organics is calculated as follows:

Aqueous Samples:

$$C_s\ (mg/L) = \frac{A_x \times D}{RF \times V_s}$$

Where:

C_s = Concentration of Gasoline Range Organics

A_x = Response for the Gasoline Range Organics in the sample, units in area

RF = Response Factor from CCS (see 9.8.4)

V_s = Volume of sample purged, in liters.

D = Dilution factor, if dilution was performed on the sample prior to analysis. If no dilution was made, D = 1, dimensionless.

Solid samples (methanol extraction):

$$C_S\ (mg/Kg) = \frac{A_x \times V_t \times D}{RF \times W \times V_i}$$

Where:

V_t = Volume of total extract (uL) (use 10000 uL for standard 10 mL extract volume).

V_i = Volume of extract actually purged (uL)

W = Weight of sample extracted, Kg. The wet weight is used.

A_x, RF, and D have the same definition as above.

Note: Some chromatographic software programs are capable of performing these calculations with minimal analyst intervention.

9.12.2 Moisture Determination (%)

Moisture (%) = (A-C)/(A-B) x 100

Where:

A = weight of aluminum boat + wet sample

B = weight of boat

C = weight of boat + dry sample

10. Quality Control (See summary Table 4)

10.1 The laboratory must, on an ongoing basis, demonstrate through the analysis of quality control check standards that the operation of the measurement system is in control. This should include the analysis of QC check samples plus the calculation of average recovery and the standard deviation of the recovery as outlined in Method 8000, Section 8.0.

10.2 After successful calibration (Section 9.3), analyze a Surrogate Control Sample. This standard is also the reagent blank sample and is analyzed with every analytical batch or every 12 hours, whichever is more frequent. The surrogate recovery should be within established limits (see appendix), or within the limits established by the project plan (whichever is more stringent).

10.3 Every batch or 20 samples, duplicate Laboratory Control Samples must be analyzed. The matrix for these samples should be reagent water for batches of aqueous samples or methanol/Ottawa sand (or other appropriate standard soil) for soil sample batch analyses. The accuracy and precision of the duplicates must be within established limits (see appendix).

10.4 With every batch of samples extracted, the methanol (reagent) blank must be analyzed. The reagent blank should have GRO less than detection limit.

10.5 If any of the criteria in 9.3, 10.2 and 10.3 are not met, the problem must be corrected before samples are analyzed.

10.6 Calculate the surrogate standard recovery in each sample. If recoveries are outside established limits, verify calculations, dilutions and standard solutions. Verify instrument performance.

10.6.1 High recoveries may be due to a coeluting matrix interference- examine the sample chromatogram.

10.6.2 Low recoveries may be due to adsorption by the sample matrix (i.e., high humus soils).

10.6.3 Low recoveries may be due to a poor purge (clogged purge tube or frit). If this is suspected, check the purge tube with a blank before reanalyzing the sample.

10.6.4 If the surrogate recovery is outside established limits due to suspected matrix effects, GRO results must be flagged. If, when adjusted for surrogate recovery, the normalized data would fall within a factor of 2 of the action limit, the laboratory should recommend that the client resubmit the sample for matrix spike/matrix spike duplicate analysis. This is a recommendation, not a requirement of the method, and therefore the onus is not on the analytical laboratory to absorb the cost of the additional analyses.

10.7 Field blanks, travel blanks, bottle blanks, duplicates, and matrix spikes are recommended for specific sampling programs.

10.8 Minimum quality control acceptance criteria are set forth in section 10 of this method. More stringent quality control criteria may be required by specific project plans.

11. Method Performance

11.1 Single-lab method performance data for the methanol extraction method in Ottawa Sand and other soil types is presented below.

11.2 Results for gasoline spikes (Methanol extraction purge and trap, soils)

Matrix	Gasoline Spike Amount mg/Kg	Percent Recovery
Ottawa Sand[1]	50	70
Ottawa Sand[1]	500	78
Houston Black Clay[1]	50	68
Houston Black Clay[1]	50	66
Norwood Loam[1]	50	60
Norwood Loam[1]	50	57
Ottawa Sand[2]	50	97
Ottawa Sand[2]	50	96
Marine Sand[2]	50	94
Glacial Clay[2]	50	68
River Sediment[2]	50	53
Marine Sediment[2]	50	132
Forest Loam, muskeg, tundra[2,3]	50	28

1 Analyses performed by Rocky Mountain Analytical. Gasoline used = API PS6.
2 Analyses performed by State of Alaska, DEC Laboratory. Gasoline used = GCS.
3 All highly organic soil matrices showed less than 30% analyte recovery.

11.3 The method detection limit calculated according to 40 CFR, Part 136, Appendix B was 0.5 mg/Kg GRO as gasoline for the methanol extraction of soils and .01 mg/L GRO as gasoline for waters. The recommended Practical Quantitation Limit (PQL) is 5 mg/Kg GRO as gasoline for soil and 0.1 mg/L GRO as gasoline for water.

12. References

1. USEPA "SW-846 Test Methods for Evaluating Solid Waste", 3rd Edition; Methods 5030, 8000, 8015 and 8020.

2. "Sampling and Analysis of Gasoline Range Organics in Soils," American Petroleum Institute Pub. #4516, October 1991.

3. "Evaluation of Proposed Analytical Methods to Determine Total Petroleum Hydrocarbons in Soil and Groundwater" prepared by Midwest Research Institute for USEPA Office of Underground Storage Tanks, August 14, 1990.

4. Urban, M.J., J.S. Smith, E.K. Schultz, R.K. Dickson, "Volatile Organic Analysis for a Soil, Sediment or Waste Sample" in Fifth Annual Waste Testing and Quality Assurance Symposium; USEPA, July 24-28, 1989.

5. Siegrist, R.L., and P.D. Jenssen, "Evaluation of Sampling Method Effects on Volatile Organic Compound Measurements in Contaminated Soils", Environmental Science and Technology, Vol. 24, November 1990.

6. Fitzgerald, John "On-site Analytical Screening of Gasoline Contaminated Media Using a Jar Headspace Procedure" in Petroleum Contaminated Soils, Vol. 2, 1989.

7. Senn, R.B., and M.S. Johnson, "Interpretation of Gas Chromatographic Data in Subsurface Hydrocarbon Investigations, "Ground Water Monitoring Review, 1987.

8. Hughes, B.M., D.E. McKenzie, C.K. Trang, L.S.R. Minor, "Examples of the Use of and Advanced Mass Spectrometric Data Processing Environment for the Determination of Sources of Wastes" in Fifth Annual Waste Testing and Quality Assurance Symposium; USEPA, July 24-28, 1989.

9. "Laboratory Study on Solubilities of Petroleum Hydrocarbons in Groundwater," American Petroleum Institute Pub #4395, August 1985.

10. "Volatile Organic Analysis for a Soil, Sediment or Waste Sample (The Methanol Method)," a symposium prepared by Dr. James S. Smith for the State of Alaska, Department of Environmental Conservation, Underground Storage Tank/Leaking Underground Storage Tank program, August 16, 1993.

APPENDIX

TABLE 1
PURGE AND TRAP OPERATING PARAMETERS
For GRO/602/8020

Parameter	Setting
Purge Gas	Nitrogen or Helium
Purge Gas Flow Rate (mL/min.)	40
Purge Time (min.)	12.0 ± 0.1
Purge Temperature (°C)	Ambient
Desorb Temperature (°C)	180
Back flush Inert Gas Flow (mL/min.)	20-60
Desorb Time	4
Trap Bake-out time	10 min.

TABLE 2
QUANTITY OF METHANOL EXTRACT NEEDED FOR ANALYSIS OF SOILS/SEDIMENTS

Approximate Concentration, GRO (mg/Kg)[a]	Volume of Methanol Extract (uL)[b]
5-100	100
200	50
1000	10
5000	100 uL of 1/50 dilution[c]

Calculate appropriate dilution factor for concentrations exceeding this table.

a This number is determined by sample pre-screening.

b The volume of methanol added to 5 mL of water being purged should be kept constant. Therefore, add to the 5-mL syringe whatever volume of methanol is necessary to maintain a total volume of 100 uL of methanol for each blank, sample and control.

c Dilute an aliquot of the methanol extract and then take 100 uL for analysis.

TABLE 3
MAJOR COMPONENTS OF API PS-6 GASOLINE

Compound	Percent Weight
2-Methylbutane	8.72
m-Xylene	5.66
2,2,4-Trimethylpentane	5.22
Toluene	4.73
2-Methylpentane	3.93
n-Butane	3.83
1,2,4-Trimethylbenzene	3.26
n-Pentane	3.11
2,3,4-Trimethylpentane	2.99
2,3,3-Trimethylpentane	2.85
3-Methylpentane	2.36
o-Xylene	2.27
Ethylbenzene	2.00
Benzene	1.94
p-Xylene	1.72
2,3-Dimethylbutane	1.66
n-Hexane	1.58
1-Methyl, 3-Ethylbenzene	1.54
1-Methyl, 4-Ethylbenzene	1.54
3-Methylhexane	1.30

Reference (9)

TABLE 4
ACCEPTANCE CRITERIA FOR QUALITY CONTROL

Analyte	Spike Concentration		Control Limits	
Lab Control Samples	Water mg/L	Soil mg/Kg	% Recovery	Relative % Difference
Gasoline Range Organics	0.5	25	60-120	20
Surrogate Control Samples				
Trifluorotoluene or Bromofluorobenzene	0.05	2.5	60-120	
Field Sample Surrogate Recovery				
Trifluorotoluene or Bromofluorobenzene	0.05	2.5	50-150	
Continuing Calibration/Laboratory Control Standards				
See 7.3	1.0	40	75-125	

The Quality Control Criteria listed in this appendix represent the minimum acceptable levels, using highly organic soil matrices. Higher performance may be required on some projects.

METHOD FOR DETERMINATION OF DIESEL RANGE ORGANICS

1. Scope and Application

1.1 Objectives

1.1.1 This method is designed to measure the concentration of diesel range organics in water and soil. This corresponds to an n-alkane range from the beginning of C_{10} to the beginning of C_{25}, and a boiling point range of approximately 170° C to 400° C.

1.1.2 The method is primarily designed to measure diesel or other mid-range petroleum products such as fuel oil. Components greater than C_{24} present in products such as motor oils or lubricating oils are detectable under the conditions of the method. More detailed identification and characterization of mid-range non-petroleum products and heavier petroleum products may be based on comparison against additional reference materials using pattern recognition techniques. These additional efforts are not specifically contained within this method.

1.2 Quantitation Limits

Practical quantitation limits (PQL) for this method for analysis of diesel range organics are based on 100 ug/mL of diesel #2 in the extract and are approximately 0.10 mg/L for waters and 4.0 mg/kg for soils.

1.3 Dynamic Range

Dilutions should be performed as necessary to put the chromatographic envelope within the linear range of the method. Linear range is dependent in part upon column type, detector sensitivity and injection volume. Typically, the approximate range is 0.01 mg/L to 100 mg/L.

1.4 Experience

This method is based on a solvent extraction, gas chromatography (GC) procedure. This method should be used by, or under the supervision of, analysts experienced in the use of solvent extractions and gas chromatographs. The analysts should be skilled in the interpretation of gas chromatograms and their use as a quantitative tool.

2. Method Summary

2.1 One liter of water or 25 grams of soil is spiked with a surrogate compound and extracted with methylene chloride. The extract is dried and concentrated to a volume of 1.0 mL. An aliquot of the extract is injected into a capillary column gas chromatograph equipped with a flame ionization detector (FID). Quantitation is performed by comparing the total chromatographic area between the peak start of C_{10} and the peak start of C_{25}, both resolved and unresolved peaks, to the response of the diesel calibration standard.

2.2 This method is primarily a modification of the American Petroleum Institute consensus "Method for the Determination of Diesel Range Organics, Revision 2, 2/5/92 [11], supplemented with information gathered by the State of Alaska, Department of Environmental Conservation, Division of Environmental Quality, Juneau Environmental Analysis Laboratory. It is based in part on US Environmental Protection Agency Methods 8000 and 8100, SW-846, "Test Methods for Evaluating Solid Waste," 3rd Edition [1], Method OA-2 [2] and work by the EPA Total Petroleum Hydrocarbons Method Committee [3], and the State of Oregon, "Total Petroleum Hydrocarbon Methods" QAR 340-122-350 dated December 11, 1990.

3. Definitions

3.1 Diesel Range Organics (DRO): All chromatographic peaks, both resolved and unresolved, eluting between the peak start of n-decane (C_{10}) and the peak start of n-pentacosane (C_{25}). Quantitation is based on direct comparison of the area within this range to the total area of the diesel standard as determined from FID response using baseline-baseline integration.

3.2 Diesel Calibration Standard (DCS): A blend of equal volumes of arctic diesel, diesel #1 and diesel #2 (1:1:1), diluted to appropriate concentrations. In those areas where arctic diesel is unavailable, kerosene may be used to prepare the calibration standard. This deviation must be noted on the final report. This standard serves as a calibration standard for diesel range organics.

3.3 Surrogate Control Standard (SCS): Ortho-terphenyl or equivalent, used as a laboratory control.

3.4 Surrogate Control Sample : A reagent water or Ottawa sand (or other standard soil, as appropriate) method blank sample spiked with SCS. The surrogate recovery is used as a laboratory control (see Appendix).

3.5 Laboratory Control Standard (LCS): A commercially prepared, certified diesel quality control standard (ERA Certified, or equivalent) used by the laboratory as a quality control check to verify the accuracy of the external calibration.

Alternatively, a NIST traceable Normal Alkane Standard (synthetic organic standard) composed of the same compounds as listed in 3.8 may be used (see Appendix).

3.6 Laboratory Control Sample: A reagent water or method blank sample spiked with a commercial diesel fuel other than those blended to make the DCS (3.2). This is used as a quality control check. The spike recovery is used to evaluate method control (see Appendix).

3.7 Pattern Recognition Standards: Various commercial petroleum products used by the laboratory to identify specific petroleum product types.

3.8 N-Alkane Standard (NAS): A mixture of normal alkanes from decane through pentacosane (C_{10} through C_{25}). This standard serves to verify expected boiling point range for petroleum products, provide data on column performance, and define the retention time window for Diesel Range Organics.

3.9 Internal Standard: Alpha androstane, used to normalize DRO concentrations. Use of an internal standard is recommended, but not required.

3.10 Standard Soil: Ottawa sand, Norwood loam, Houston black clay or other standard soil with characteristics which match the field samples as closely as possible, used in quality control standards.

3.11 Other terms are as defined in SW-846 [1].

4. Interferences

4.1 Other organic compounds including, but not limited to, animal and vegetable oil and grease, chlorinated hydrocarbons, phenols, phthalate esters and biogenic terpenes are measurable under the conditions of this method. Heavier petroleum products such as lubricating oil, and crude oils also produce a response within the retention time range for DRO. As defined in the method, the DRO results include these compounds.

4.2 Method interferences are reduced by washing all glassware with hot soapy water and then rinsing it with tap water, methanol, and methylene chloride. Heating the glassware to reduce contaminants should not be necessary if this cleaning method is followed. At least one reagent blank must be analyzed with each extraction batch to demonstrate that the samples are free from method interferences.

4.3 High purity reagents such as Burdick and Jackson GC[2] methylene chloride or Baker capillary grade methylene chloride must be used to minimize interference problems.

4.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of a solvent blank to check for instrument contamination.

5. Safety Issues

5.1 The toxicity or carcinogenicity of each reagent in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety should be available and should be identified for use by the analyst.

5.2 A hearing protection device should be used when performing sonication.

6. Apparatus

6.1 Glassware

6.1.1 All specifications are suggested only.

6.1.2 4 oz. amber glass wide mouth jars with Teflon lined screw caps

6.1.3 Separatory funnel - 2000 mL with Teflon stopcock

6.1.4 Continuous liquid-liquid extractor - equipped with Teflon or glass connecting joints and stopcocks requiring no lubrication (Hershberg-Wolf Extractor, Ace Glass Company, Vineland, New Jersey, P/N6841-10, or equivalent).

6.1.5 Concentrator tube. Kuderna-Danish 10 mL graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stopper is used to prevent evaporation of extracts.

6.1.6 Evaporative flask, Kuderna-Danish 500 mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with springs.

6.1.7 Snyder column, Kuderna-Danish three ball macro (Kontes K-503000-0121 or equivalent). Rotary evaporation set-up may be used alternatively.

6.1.8 Jars: One liter amber glass, with Teflon lined screw caps.

6.1.9 Two mL glass vials with Teflon-lined cap (autosampler vials).

6.1.10 Disposable pipettes: Pasteur.

6.1.11 Graduated cylinders: 250 mL.

6.1.12 Glass or Teflon funnels.

6.2 Boiling chips - Approximately 10/40 mesh. Heat to 400°C for 30 minutes or Soxhlet extract with methylene chloride.

6.3 Microsyringes: 1 ul, 5 ul, 10 ul, 25 ul, and 100 ul.

6.4 Water bath - Heated with concentric ring cover, capable of temperature control (+/- 2°C). The bath should be used in a hood.

6.5 An analytical balance capable of accurately weighing 0.0001 g should be used for preparing standards. A top-loading balance capable of weighing to the nearest 0.1 g should be used for sample preparation.

6.6 Stainless steel spatula.

6.6 Gas Chromatography

6.6.1 Gas Chromatograph: Analytical system including appropriate gas supply and all required accessories, including a Flame Ionization Detector (FID), column supplies, gases, and syringes. A data system capable of determining peak areas using a forced baseline - baseline projection is required. A data system capable of storing and reintegrating chromatographic data is recommended.

6.6.2 Columns

6.6.2.1 Column 1: 25 M x 0.25 mm Quadrex 007 5% methyl phenyl 0.5 micron film thickness.

6.6.2.2 Alternate column: 30 M x 0.53 mm ID Restek RTX-5, 1.5 micron film thickness.

6.6.2.3 Other Columns may be used - capillary columns are required to achieve the necessary resolution. See 9.2.2 for column performance criteria.

6.7 Sonication

6.7.1 Ultrasonic cell disrupter: A horn-type sonicator equipped with a titanium tip should be used. A Heat Systems-Ultrasonics, Inc. Model W-385 (475 watt) sonicator or equivalent (power wattage must be a minimum of 375 with pulsing capability and No. 200 1/2 inch Tapped Disrupter Horn) plus No. 207 3/4 inch Tapped Disrupter Horn, and No. 419 1/8 inch Standard tapered Microtip probe.

6.7.2 A Sonabox is recommended with the above disrupter for decreasing sound (Heat Systems-Ultrasonics, Inc. Model 432 13 or equivalent).

6.8 Soxhlet extraction apparatus as described in SW-846 Method 3540 [1].

6.9 Nitrogen evaporator with high purity (grade 4.5 or equivalent) nitrogen gas source.

7. Reagents and Standards

7.1 Reagent water: Carbon filtered deionized water that has been shown to be free from Diesel Range Organic compounds - a Millipore system or equivalent is recommended.

7.2 Methylene chloride, hexane, acetone - pesticide grade or equivalent.

7.3 Sodium sulfate - (ACS grade) granular, anhydrous. Purify by heating at 400°C for 4 hours in a shallow tray, or by extracting three times with methylene chloride and drying at 105° C. Incomplete cleaning of sodium sulfate can result in phthalate contamination of samples.

7.4 Stock standard solution - Prepare the following stock standards. Unless noted, all are prepared in the methylene chloride listed in 7.2 above. Standard preparation should follow guidelines in Method 8000 [1].

7.4.1 Optional Stock Internal Standard: 1000 ug/mL 5 alpha-androstane. Other internal standards may be used provided they do not interfere with the DRO components.

7.4.2 Recommended Surrogate Control Standard: 200 ug/mL ortho-terphenyl (OTP). A working solution is made at 20 ug/mL (recommended concentration) in acetone.

7.4.3 Diesel Calibration Standard: A blend of equal volumes of diesel fuel, mixed together to form a composite diesel fuel (1:1:1, arctic diesel : diesel #1 : diesel #2) is used to prepare standards in methylene chloride. No fewer than 3 concentrations of this DCS are used for instrument calibration. A five point calibration curve is recommended. Other than one standard concentration near the method detection limit, the expected range of concentrations found in project samples should define the working range of the GC.

7.4.4 Normal Alkane Standard (NAS): A stock solution of C_{10} - C_{25} even and odd n-alkanes with each component at a level of at least 2000 ug/mL. For solubility reasons, it may be necessary to prepare stocks of individual alkanes in other solvents such as hexane or chloroform. At a minimum, this multi-component blend of alkanes serves as a retention time window defining mix for DRO. With NIST traceable components, it may also serve as LCS (see 3.5). See table in appendix for boiling point range to be covered.

7.4.5 Stock Laboratory Control Standard - 25000 ug/mL commercial diesel #2 other than that used to prepare the Diesel Calibration Standard (7.4.3). A working solution is made at a recommended concentration of 5000 ug/mL in acetone.

8.0 Sample Collection, Preservation, Containers, and Holding Times

8.1 Water samples are collected, in duplicate, in one liter amber glass containers with Teflon lined screw caps and acidified to pH 2 or less with HCl. Soils are collected in a core tube, or 4 or 8 oz amber glass jar with Teflon lined lid. The samples are stored at 4° $\pm$ 2° C from the time of collection until extraction. Extraction must be performed on waters within 7 days and soils within 14 days. All analyses of extracts must take place within 40 days.

9. Procedure

9.1 Sample Preparation

9.1.1 Waters are extracted according to SW-846 Method 3510 (Separatory Funnel Liquid-Liquid Extraction) or Method 3520 (Continuous Liquid-Liquid Extraction). Soil samples are extracted using Method 3550 (Sonication). Method 3540 (Soxhlet Extraction) may also be used.

9.1.2 Water extraction - Separatory Funnel.

9.1.2.1 Measure a 1 L portion of the sample and transfer to a 2 L separatory funnel. If the sample is in a 1 L or smaller bottle, mark the water meniscus on the side of the sample bottle for later determination of the sample volume (see 9.1.3.5). If the sample is in a larger bottle, use a 1 L graduated cylinder to measure the volume of the sample. Use no more than 1 L of sample per 2 L separatory funnel. For blanks and quality control standards, pour 1 L of carbon filtered water into the separatory funnel.

9.I.2.2 Check and note the pH.

9.1.2.3 Add 1 mL of ortho-terphenyl surrogate standard (recommended level of 20 ug/mL).

9.1.2.4 For every batch or 20 samples extracted (whichever is more frequent), prepare duplicate Laboratory Control Samples by adding 1 mL of 5000 ug/mL LCS (7.4.5) to each of two 1 L volumes of carbon filtered water. Daily or for every 20 samples (whichever is more frequent), prepare a blank surrogate control standard using 1 L of carbon filtered, ASTM Type II organic free water.

9.1.2.5 For samples, add 60 mL methylene chloride to the sample bottle to rinse the inner walls after the sample has been transferred to the separatory funnel. **Do Not** cap and shake the bottle, rinse the glass only; transfer the solvent to the separatory funnel. Extract the sample by shaking it for no less than two minutes with frequent ventilation.

9.1.2.6 Allow the layers to separate (approx 10 minutes rest after shaking). If there is an emulsion, break it. If the emulsion cannot be broken (recovery of <80% of the methylene chloride, corrected for water solubility of methylene chloride), transfer the sample, solvent, and emulsion into the extraction chamber of a continuous extractor and proceed as described in 9.1.3. Alternative physical techniques for breaking up emulsions may be acceptable.

9.1.2.7 Drain the bottom layer (methylene chloride) into a 250 mL graduated cylinder or other calibrated glassware.

9.1.2.8 Repeat the extraction twice more, using a 60 mL aliquot of methylene chloride each time. Collect the solvent in the same graduated cylinder (or equivalent) as described in 9.1.2.7. Record the volume recovered and other prep information.

9.1.2.9 Put a plug of glass wool in a glass or teflon funnel and fill about 2/3 full with anhydrous sodium sulfate. Rinse the funnel and sodium sulfate with 30-40 mL of methylene chloride, discard rinsate. Pour the extract through the rinsed sodium sulfate into a 500 mL Kuderna-Danish (K-D) evaporative concentrator. Rinse the graduated cylinder, then the sodium sulfate, with small amounts of methylene chloride. Add these rinses to the K-D.

9.1.2.10 Add a few boiling chips to the K-D and attach a 3 ball Snyder to the top. Pre-wet the column by adding about 1 mL of methylene chloride to the inverted column before attaching it to the K-D.

NOTE: The concentration step is critical; losses of target compounds can occur if care is not taken.

9.1.2.11 Place the K-D in a heated water bath set at 95°C so that the receiver tube is immersed in hot water and the entire lower rounded surface is bathed in steam. At a proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume reaches 5-10 mL, remove the K-D from the bath and allow it to cool completely.

Note: The extraction and concentration steps must be performed under a hood. Not only is the methylene chloride a potential health hazard (see MSDS), if the heated water bath is not properly temperature controlled then the concentration apparatus can explode.

9.1.2.12 After the K-D has cooled, rinse the Snyder column and middle flask with a small amount of methylene chloride. Transfer the extract to a calibrated 15 mL centrifuge tube, rinsing with a small amount of methylene chloride. Be sure to rinse all of the ground glass joints well, as compounds collect on the ground glass.

9.1.2.13 Carefully concentrate the extract to 1.0 mL under a gentle stream of nitrogen using the evaporation apparatus. If the extract is highly colored, forms a precipitate, or stops evaporating, the final volume should be higher (5-10 mL). Transfer to a labeled 4 mL (or 12 mL) vial with Teflon lined cap, mark the meniscus. Extracts should be stored in a non-frost free freezer.

9.1.2.14 Record information for the extraction and concentration steps.

9.1.3 Water Extraction - Continuous liquid-liquid extraction (follow manufacturer's instructions).

9.1.3.1 Mount the continuous extractor on appropriate racks.

9.1.3.2 Put 250 mL methylene chloride in a round bottom flask, add a few boiling chips. Add 300 mL of methylene chloride to the extractor flask.

9.1.3.3 Check and note the initial pH of the sample. Prepare surrogate and laboratory control standards as in 9.1.2.3 and 9.1.2.4.

9.1.3.4 When introducing water into the extractor, minimize disturbance of the solvent layer and avoid getting water into either sidearm by carefully pouring the water down the back of the extractor.

9.1.3.5 For samples in 1 L or smaller bottles, mark the meniscus on the side of the sample bottle and pour approximately 1 L of the sample into the extractor flask. Measure the exact volume by adding tap water to the bottle to the marked level and measuring the volume with a graduated cylinder. For samples in larger bottles, measure 1 L of the sample in a graduated cylinder. Record the volume of sample used for extraction.

9.1.3.6 If necessary, add enough carbon free water to the extraction flask to allow the solvent in the removable side arm to just begin to drip into the round bottom flask. Record the total volume of carbon free water that was added.

9.1.3.7 Remove the condenser from the rack and wipe the lower joint and lip with a tissue soaked with solvent. Place the condenser on top of the extractor. Turn on the cool water supply and check the flow indicators.

9.1.3.8 Turn on the heating mantle and record the starting time. Check after 15 minutes to be sure that the solvent in the round bottom flask is boiling, that solvent is dripping from the lip on the condenser, and that the volume of the solvent in the round bottom flask is still about 240 mL.

9.1.3.9 Check all extractor joints for leaks with a Kimwipe. Allow the extraction to proceed for 18-24 hours. Surrogate recovery from blanks can be used as an indicator of extraction efficiency, and extraction times can be adjusted to optimize results.

9.1.3.10 Turn off the heating mantle and allow the apparatus to cool (30-60 minutes) with water flowing through the condenser.

9.1.3.11 The solvent contained in the round bottom flask is the extract. Transfer the extract to a 400 mL glass graduated cylinder, rinsing with a small amount of Methylene chloride. If the volume of solvent is less than about 250 mL, record it.

9.1.3.12 Go to 9.1.2.9 and proceed with the prep, through the nitrogen concentration.

9.1.4 Soil Preparation - Sonication

9.1.4.1 Decant any water layer which may accompany the solid layer in the sample. Note what percent of the sample the water represents and, if sufficient volume exists, extract and analyze the water for DRO. Also note the apparent condition of the sample (e.g., presence of foreign materials, variable particle size, presence of oil sheen, multiple phases, etc).

9.1.4.2 Since final numbers are reported as mg/dry Kg, a moisture determination must be made. Pre-weigh an aluminum weighing boat. Weigh 5-10 g of the sample into the boat and record both weights on a percent moisture worksheet. Dry the sample in a hood or warm (no greater than 100 °C ± 5 °C) oven overnight. If necessary, cool in a desiccator until the sample reaches room temperature and re-weigh for the moisture determination.

$$\% \text{ Moisture} = (A-C)/(A-B) \times 100$$

Where: A = weight of boat + wet sample

B = weight of boat

C = weight of boat + dry sample

Note: Make sure drying oven is placed under a hood. Heavily contaminated soils will produce strong organic vapors which may be detrimental to analysts' health.

9.1.4.3 Weigh 25 g of the original sample into a 250 mL centrifuge bottle and record the weight to the nearest 0.001 g. Add 25 g of dried sodium sulfate and stir the mixture well with a steel spatula (do **not** use plastic). The sample should have a grainy texture. If it forms a large clump, continue adding sodium sulfate until the grainy texture is achieved and note the total weight of dehydrant used.

9.1.4.4 Add 100 mL of Methylene chloride to all samples.

9.1.4.5 Add 1 mL of 200 ug/mL ortho-terphenyl to all samples and controls. Make sure that the pipet tip is below the surface of the solvent while the standard is being added. Mix the samples immediately.

9.1.4.6 Add one mL of 5000 ug/mL LCS to the duplicate laboratory control samples. These standards should contain 25 g of the appropriate standard soil, pre-extracted with methylene chloride and dried before weighing. In addition, prepare a reagent blank/surrogate control standard using 25 g of standard soil and containing 1 mL of 200 ug/mL ortho-terphenyl.

9.1.4.7 Sonicate the samples for 3 minutes at an output setting of 10 with the 3/4 inch sonicator horn 1/2 inch below the surface of the solvent. The sonicator should be in the 1 second pulse mode, with the duty cycle set at 50%.

9.1.4.8 Decant and filter the extracts through Whatman No. 1 filter paper (or other, non-interacting filter paper), using vacuum or pressure filtration, into a solvent rinsed 500 mL graduated cylinder. Use caution to not tear the wetted filter. Alternately, the extracts may be centrifuged (recommend 35 rpm) and decanted.

9.1.4.9 Repeat the extraction twice more using 100 mL aliquots of methylene chloride each time. Collect these extracts in the same cylinder described in 9.1.4.8.

9.1.4.10 Record the total volume of the solvent that is recovered.

9.1.4.11 Go to 9.1.2.9 and proceed.

9.1.5 Soil Preparation - Soxhlet Extraction

9.1.5.1 Decant any water layered on the sample. Mix the sample well and note any foreign objects or anomalies (see 9.1.4.1).

9.1.5.2 Weigh 10 g to 30 g of the original sample into an extraction thimble. Add an equal weight of anhydrous sodium sulfate and stir the mixture well with a wooden tongue depressor. The sample should have a grainy texture - if the sample clumps, add more sodium sulfate until a grainy texture is achieved and note the addition. (Do this for all samples and standards.) Do a moisture determination (see 9.1.4.2).

9.1.5.3 Place loaded thimbles in extractors and add 1 mL of surrogate to all samples and standards.

9.1.5.4 Add one mL of LCS to the duplicate laboratory control samples. These standards should contain 10 g of methylene chloride rinsed Ottawa Sand or alternative standard soil. In addition, prepare a reagent blank surrogate control standard containing 1 mL of ortho-terphenyl.

9.1.5.5 Add 300 mL of methylene chloride to the 500 mL extraction flask. Also add a few methylene chloride washed carborundum boiling chips to the flask. Connect the extractor to the flask and the condenser to the extractor. Allow samples to extract for 18-24 hours, or as long as necessary to achieve optimum surrogate recovery. Be sure that coolant is flowing around the condensers.

9.1.5.6 After extraction, disassemble extractor and add about 3 g anhydrous sodium sulfate to the extract and allow to incubate for 2 hours. (This assures that the extract is water-free before concentration.)

9.1.5.7 Transfer extract into a clean 500 mL K-D and proceed from 9.1.2.9.

9.1.6 Dilution Technique

9.1.6.1 This is used for product or waste samples for which extraction is not appropriate, and which are soluble in methylene chloride.

9.1.6.2 Weigh 1 g of sample into a 10 mL volumetric flask. Dilute to 10 mL with methylene chloride. Transfer to a 12 mL vial with a teflon lined lid. Mark meniscus and store at 4 (± 2) °C.

9.2 Gas Chromatography

9.2.1 Conditions (Recommended):

Set helium column pressure to 20#. Set column temperature to 40° C for 2 minutes, then ramp at a rate of 12° C/min to 320° C and hold for 15 min. (run time = 36 minutes). Set FID Detector to 320° C and injector to 280° C. The reference book High Resolution Chromatography by Hewlett-Packard is a good source of information on how to optimize flow rates, etc.

9.2.2 Performance Criteria: GC run conditions and columns must be chosen to meet the following criteria:

9.2.2.1 Resolution of the methylene chloride solvent front from C_{10}.

9.2.2.2 Area of C_{24} within 20% of area of C_{17}. (Mass discrimination check).

9.2.2.3 The separation number, TZ, should be greater than 15 for the dodecane/tridecane paraffins.

$$TZ = \frac{\text{retention time tridecane - retention time dodecane}}{W_{1/2} \text{ of tridecane} + W_{1/2} \text{ of dodecane}} - 1$$

Where "$W_{1/2}$" = peak width at half-height

9.2.2.4 The column must be capable of separating typical diesel components from the surrogate and internal standards. In particular, there are potential problems with the resolution of n-C_{19}/ortho-terphenyl and n-C_{21}/5 alpha-androstane at varying relative concentrations.

9.3 Calibration

9.3.1 Calibrate the GC with an initial five point (recommended) calibration using DCS (7.4.3). The final calibration curve must be represented by no less than 3 concentrations of DCS. Tabulate the area response of the diesel against mass injected. The ratio of the response to the amount injected,

defined as the response factor (RF), can be calculated for the standard at each concentration. If the average percent relative standard deviation (%RSD) is less than 25% over the working range, linearity through the origin can be assumed, and the continuing calibration response factor can be used in place of a calibration curve.

$$RF = \frac{\text{Total area of diesel standard x Internal standard amount(mg/mL)}}{\text{Diesel standard amount (mg/mL) x Internal standard area}}$$

Alternately, external standard calibration may be used (See Method 8000 [1]). Then,

$$RF = \frac{\text{Total area of diesel standard}}{\text{DCS amount (mg/mL)}}$$

9.3.2 The calibration curve must be confirmed using the LCS. This certified (or traceable) standard verifies the accuracy of the calibration. The concentration of the LCS should be within the expected concentration range of the samples to be analyzed.

9.3.3 The working RF or calibration curve must be verified on each working day by the injection of a continuing calibration standard (CCS) at a concentration mid-point on the calibration curve. If the response for this standard varies from the predicted response by more than 25%, a new calibration curve must be prepared.

$$\text{Percent Difference} = \frac{R1 - R2}{Ravg} \times 100$$

where:
R1 = Average RF from the calibration curve
R2 = Response Factor from CCS
Ravg = (R1 + R2)/2

9.4 Retention Time Window Definition (Note: Although the retention time window definition may introduce a bias, it improves precision and reduces interferences from most other petroleum products.)

9.4.1 Before establishing windows, be certain that the GC system is within optimum operating conditions (6.6). Make three injections of the NAS (7.4.3) throughout the course of a 72 hour period. Serial injections over less than a 72 hour period result in retention time windows that are too tight.

9.4.2 Calculate the standard deviation of the three absolute retention times for each NAS component (decane and tetracosane if NAS is used as a retention time window standard only, all NAS components if it is to be additionally used as LCS) and the surrogate.

9.4.2.1 The retention time window for individual peaks is defined as plus or minus three times the standard deviation of the absolute retention time for each component.

9.4.2.2 In those cases where the standard deviation for a particular analyte is zero, the laboratory should use ± 0.05 min. as a retention time window.

9.4.3 The laboratory must calculate retention time windows for each standard on each GC column and whenever a new GC column is installed or instrument conditions changed. The data must be retained by the laboratory for at least a year.

9.4.4 Retention time windows must be verified regularly and updated no less frequently than once a year.

9.5 Gas Chromatograph Analysis

9.5.1 Samples are analyzed by GC/FID. Optimum injection volumes (2 uL using the conditions established in 9.2) must be established for specific instrument conditions.

9.5.2 For internal standard calibration, the internal standard is spiked into each sample and standard at a concentration of 200 ug/mL of sample extract. Twenty uL of 5-alpha androstane stock at 1000 ug/mL may be spiked into the 1 mL final volume or a corresponding amount may be added to an aliquot of the final extract. (Note: Diesel range organic values >2000 ug/mL may lead to measurement bias due to coelution with the internal standard.)

9.5.3 If initial calibration (9.3.1) has been performed, verify the calibration by analysis of a mid-point CCS (9.3.2). With each day's run, open a 24 hour analysis window. This is done by running the NAS.

9.5.4 Calculate the percent difference of the response factor from the mean response factor as in 9.3.2. This is done for diesel range organics as a group from the CCS if DRO only is to be quantitated, and for each of the NAS components if additional quantitation is required. If the response factors have a percent difference greater than 25%, the instrument must be recalibrated. (9.3.1)

9.5.5 The midpoint LCS must also be run once every twenty samples and at the end of each sequence. If the result does not fall within 25% of the true value, all samples run before the failed QC must be reanalyzed.

9.5.6 A methylene chloride blank must be run in every sequence to determine the area generated on normal baseline bleed under the conditions prevailing in the 24 hour period. This area is generated by projecting a horizontal baseline between the retention times observed for the peak start of C_{10} and the peak start of C_{25}. This laboratory control sample is integrated over the DRO area in the same manner for the field samples, and is reported as the solvent blank. (Refer to reference 4.) **Do not baseline subtract. This information is for data interpretation purposes only.**

9.5.7 Methylene chloride blanks should also be run after samples suspected of being highly concentrated to prevent carryover. If the blank analysis shows contamination, the column must be baked out and subsequent blanks analyzed until the system is shown to be free from contaminants.

9.5.8 If the product concentration exceeds the linear range of the method (as defined by the range of the calibration curve) in the final extract, the extract must be diluted and reanalyzed. The response of the major peaks should be kept in the upper half of the linear range of the calibration curve. Due to potential measurement bias, internal standard calibration should not be used when DRO exceeds 5000 ug/mL in the final extract. The sample should be diluted or external standard calibration should be used.

9.5.7 Qualitative identification (Pattern Recognition):

9.5.7.1 Identification of diesel or other products is achieved by direct comparisons of sample chromatograms to retention times and peak patterns of standard product chromatograms. Regardless of whether a product identification can be made on this basis, an alkane range or boiling point range should be reported to encompass approximately 95% of the product envelope, along with the midrange boiling point of the area of greatest response.

9.5.7.2 The analyst should generate a value for diesel range organics **and** qualitatively identify diesel or other products when reporting data. The experience of the analyst weighs heavily in the interpretation of the chromatogram. References 7, 8, 9 and 10 contain some background information on hydrocarbon pattern recognition. Environmental samples may contain more than one type of product, and loss of light end components may mean the product has been in the subsurface a longer period of time.

9.5.7.3 For qualitative information, a footnote should be added. This footnote should include an alkane range and, if possible, product identification.

9.6 Calculations:

9.6.1 Internal Standard Calibration: The concentration of Diesel Range Organics in the sample is determined by calculating the absolute weight of analyte chromatographed from a summation of peak response for all chromatographic peaks eluting between the peak start of n-decane and the peak start of n-pentacosane, using the calibration curve or the response factor determined in section 9.3. Also refer to Section 9.4 (Retention Time Window Definition). The concentration of Diesel Range Organics is calculated as follows:

Aqueous/Soil samples:

$$Cs = \frac{Ax \times Cis \times Vt \times D}{As \times RF \times Vs}$$

Where:

Cs = Concentration of Diesel Range Organics (mg/L or mg/Kg).

Ax = response for the Diesel Range Organics in the sample, units in area.

RF = Response Factor from CCS (see 9.3.1).

As = Response for the internal standard, units same as for Ax.

Cis = Internal standard concentration (mg/mL).

Vt = Volume of Final extract in mL.

D = Dilution factor, if dilution was performed on the sample prior to analysis. If no dilution was made, D = 1, dimensionless.

Vs = Amount of sample extracted in L or Kg.

9.6.2 In order to calculate mg/dry Kg for soil samples,

$$\text{mg/dry Kg DRO} = \frac{Cs}{1-(\%\ \text{moisture}/100)}$$

The % moisture calculation should be included in the data package (see 9.1.4).

9.6.3 External Standard Calibration:

Aqueous/Soil samples:

$$Cs = \frac{Ax \times A \times Vt \times D}{As \times Vs}$$

Where:

Cs = Concentration of Diesel Range Organics (mg/L or mg/Kg).

Ax = response for the Diesel Range Organics in the sample, units in area.

As = Response for the external standard, units same as for Ax.

A = External standard concentration (mg/mL).

Vt = Volume of Final extract in mL.

D = Dilution factor, if dilution was performed on the sample prior to analysis. If no dilution was made, D = 1, dimensionless.

Vs = Amount of sample extracted in L or Kg.

10. Quality Control

10.1 The laboratory must establish and maintain the ability to generate acceptable accuracy and precision, and to demonstrate through the analysis of quality control check standards that the operation of the measurement system is in control. This should include the analysis of QC check samples plus the calculation of average recovery and the standard deviation of recovery as outlined in method 8000, section 8.0 [1].

10.2 After successful calibration (Section 9.8), analyze a Surrogate Control Sample. This standard is also the reagent blank sample and is analyzed with every analytical batch (20 samples) or sequence, whichever is more frequent. The surrogate recovery should be within established limits (see Appendix), or within the limits established by the project plan (whichever is more stringent) and the control sample should not have Diesel Range Organics above the practical quantitation limit.

10.3 Every batch or 20 samples, duplicate Laboratory Control Standards must be analyzed (organic free water or Ottawa sand matrix, as is appropriate to the samples being analyzed). The accuracy and precision of the duplicate standards must be within established limits.

10.4 Every batch of samples extracted must be accompanied by a reagent blank to demonstrate that samples are free from method interference.

10.5 Each laboratory should generate control limits based on the average recovery, with ± 2 standard deviations as a warning limit and ± 3 standard deviations the action limit.

10.6 If any of the criteria in 9.3, 10.2 and 10.3 are not met, the problem must be corrected before samples are analyzed.

10.7 Calculate the surrogate standard recovery in each sample. If recoveries are outside established limits verify calculations, dilutions and standard solutions. Verify instrument performance.

10.6.1 High recoveries may be due to a coeluting matrix interference or the presence of high molecular weight contaminants; examine the sample chromatogram.

10.6.2 High recoveries may also be due to memory effects caused by poor sample volatility, backflash or carryover; check instrument conditions, injection volume and injector temperature.

10.6.3 Low recoveries may be due to adsorption by the sample matrix (muskeg, tundra, forrest loam, etc).

10.6.4 Low recoveries may also be caused by incorrect integration. The chromatographic profile of diesel oils does not give baseline resolution of all components, resulting in a characteristic rise in baseline underneath the resolved hydrocarbon components. Do not use peak to peak integration, use forced baseline integration.

10.6.5 If internal calibration has been used, DRO results must be normalized using the internal standard response. If surrogate recovery is still outside of established limits, the results must be flagged and an explanation offered.

10.6.6 If external calibration has been used, and surrogate recovery is outside of established limits, the results must be flagged and an explanation offered.

10.7 If, when field samples show low surrogate recovery due to suspected matrix interference and the normalized Diesel Range Organics concentration falls within a factor of 2 of the action level, the laboratory should recommend that the client resubmit the sample for matrix spike/matrix spike duplicate analysis. (To perform matrix spike analyses, follow 9.1, except use a field sample instead of a standard matrix.) This is a recommendation, not a requirement, of the method and therefore the onus is not on the analytical laboratory to absorb the cost of the additional analyses.

10.8 Field blanks, travel blanks, matrix blanks, duplicates and matrix spikes are recommended for specific sampling programs.

10.9 Minimum quality control acceptance criteria are set forth in section 10 of this method. More stringent quality control criteria may be required by specific project plans.

11. Method Performance

11.1 Single lab method performance data for the diesel range organics method in Ottawa sand and other soil types is presented below.

11.2 Results for diesel spikes (methylene chloride extraction direct injection, soils)

Matrix	Diesel Spike Amount mg/Kg	Percent Recovery
Ottawa Sand	70	97
Ottawa Sand	70	98
Glacial Blue Clay	70	70
Glacial Blue Clay	70	76
Forrest Loam	70	136
Forrest Loam	70	163
River Sediment	70	142
River Sediment	70	167
Marine Sand	70	95
Marine Sand	70	88

Analyses performed by State of Alaska, DEC Laboratory. Diesel used = DCS.
All highly organic soil matrices showed nigh analyte recovery due to naturally occurring diesel range organics.

11.3 The method detection limit for soil calculated according to 40 CFR, Part 136, Appendix B was 1.6 mg/Kg (external standard calibration, Ottawa sand).

12. References

1. USEPA "SW-846 Test Methods for Evaluating Solid Waste", 3rd Edition; Methods 8000, 8100, 3510, 3520, 3540, and 3550.

2. "Method OA-2: Extractable Petroleum in Products," Revision January 10, 1990", University Hygienic Laboratory, Iowa City, Iowa.

3. "Method for Determination of Extractable Petroleum Hydrocarbons (EPH) in Soil and Water," Draft-February 28, 1990, prepared for Total Petroleum Hydrocarbons Method Committee by Midwest Research Institute.

4. Zilis, K., M. McDevitt, and J. Parr, "A Reliable Technique for Measuring Petroleum Hydrocarbons in the Environment," presented at the conference on Petroleum Hydrocarbons and Organic Chemicals in Groundwater, NWWA, Houston, Texas, November 1988.

5. American Petroleum Institute "Method for the Determination of Diesel Range Organics," Draft Revision 2-February 5, 1992, prepared for Total Petroleum Hydrocarbons Method Committee.

6. "Leaking Underground Fuel Tank (LUFT) Field Manual, "State Water Resources Control Board, State of California, Sacramento, CA, May 1988.

7. Fitzgerald, John, "Onsite Analytical Screening of Gasoline Contaminated Media Using a Jar Headspace Procedure" in Petroleum Contaminated Soils, Vol. 2, 1989.

8. Senn, R.B., and M.S. Johnson, "Interpretation of Gas Chromatographic Data in Subsurface Hydrocarbon Investigations," Ground Water Monitoring Review, 1987.

9. Hughes, B.M., and D.E. McKenzie, C.K. Trang, L.S.R. Minor, "Examples of the Use of an Advanced Mass Spectrometric Data Processing Environment for the Determination of Sources of Wastes" presented at 5th Annual Waste Testing and Quality Assurance Symposium, July 24-28, 1989.

10. ASTM "Standard Methods for Comparison of Waterborne Petroleum Oils by Gas Chromatography," pp. 3328-78.

11. API consensus "Method for the Determination of Diesel Range Organics," Revision 2, 2/5/92.

12. Research done by the State of Alaska, Department of Environmental Conservation, Division of Environmental Quality, Juneau Environmental Analysis Laboratory.

APPENDIX

NAS COMPONENT STANDARD
NORMAL ALKANES AND BOILING POINTS
(Reference use only)

	Compound	Boiling Point
C_{10}	Decane	174
C_{12}	Dodecane	216
C_{14}	Tetradecane	254
C_{16}	Hexadecane	287
C_{18}	Octadecane	316
C_{20}	Eicosane	344
C_{22}	Decosane	369
C_{24}	Tetracosane	391
C_{26}	Hexacosane	412
C_{28}	Octacosane	431

From "Manual on Hydrocarbon Analysis," ASTM Committee D-2 on Petroleum Products and Lubricants, Third Edition, 1977.

ACCEPTANCE CRITERIA FOR QUALITY CONTROL

(Or, as described the Quality Assurance Project Plan, whichever is most stringent)

Analyte	Spike Concentration		Control Limits	
Lab Control Samples	Water (mg/L)	Soil (mg/kg)	Relative % Recovery	% Difference
Diesel Range Organics	0.5	20	60 - 120	20
LCS/CCS				
Diesel Range Organics	2.5	100	75 - 125	
Surrogate Control Samples				
ortho-terphenyl	0.02	0.8	60 - 120	
Surrogate Recovery (field samples)				
ortho-terphenyl	0.02	0.8	50 - 150	

mj/pp/kkp(share\sop\AK102)

MILLIPORE

EnviroGard™ PCB Test Kit

ENVR 000 09 (with PCB calibrators)
ENVR 0NC 09 (*without PCB calibrators*)

Intended Use

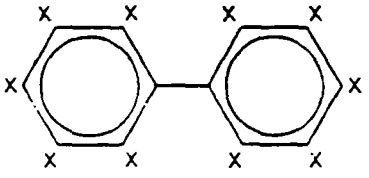
The Millipore™ EnviroGard PCB Test Kit is an enzyme immunoassay for the detection of a range of polychlorinated biphenyls (PCB) in soil, to include Aroclors 1016, 1242, 1248, 1254, and 1260. The EnviroGard PCB Test Kit allows for reliable and rapid screening for PCB in soils at specified action levels within the following sensitivity ranges:

- 5, 10, 50 part per million (ppm) (standard range)
- 1, 5, 10 ppm (high sensitivity range)

NOTE: If you use the EnviroGard PCB test kit without calibrators (ENVR 0NC 09), modify the directions according to the calibrator or standards in use.

Test Summary and Explanation

PCBs are a family of compounds with the following general structure.



where X = Hydrogen (H) or Chlorine (Cl)

There are 209 individual forms (or congeners) containing from 1–10 chlorine atoms on the biphenyl structure shown. PCBs were originally sold in the U.S.A. under the trade name Aroclor. Each Aroclor is composed of many congeners. Many Congeners may appear in more than one Aroclor. Aroclors are differentiated on the basis of average chlorine content (percent chlorine by weight). For Aroclor nomenclature, the last two digits of the four digit label indicate this percentage. For example, Aroclor 1248 is approximately 48% chlorine by weight. The sole exception to this rule, Aroclor 1016, is similar in total chlorine content to Aroclor 1242, but contains a different congener distribution.

NOTE: Refer to the section "Results Interpretation" and "Specificity" for more information on Aroclors.

The EnviroGard PCB Test Kit employs an antibody against PCB that is coated onto 12 X 75 millimeter (mm) polystyrene test tubes. The method is based on the principles of competitive immunoassay, where the absorbance signal (optical density) of the final reaction mixture is inversely proportional to the concentration of analyte (PCB) present in the original sample. A soil sample that generates a signal greater than the signal of the PCB assay calibrator (e.g., 50 ppm) has a 99% probability of containing less PCB than the specified assay calibrator (e.g., < 50 ppm).

Test Principles

PCBs present in soil extracts and assay calibrators are bound during the first incubation by the anti-PCB antibodies, which have been adsorbed onto the test tubes. After you decant the sample and wash test tubes, a peroxidase-PCB conjugate is added.

NOTE: The amount of conjugate that is bound (by unoccupied anti-PCB antibody binding sites in the test tube) is inversely proportional to the amount of PCB that was originally present in the sample.

After a 5-minute incubation, unbound conjugate is decanted and the test tubes are washed again. Finally, a solution that contains a chromogenic peroxidase substrate is added to the test tubes.

NOTE: Color development is directly proportional to enzyme concentration and inversely proportional to PCB concentration in the original sample in the test tubes.

The determination of PCB level in unknown samples is interpreted relative to standard assay calibrator levels (e.g., 1, 5, 10, 50 ppm) or Aroclor standards, using visual comparison or reading by a spectrophotometer.

Precautions

- Treat PCBs, solutions that contain PCBs, and potentially contaminated soil samples as hazardous materials.
- Use gloves, proper protective clothing, and means to contain and handle hazardous material where appropriate.
- Obtain (if appropriate) permits pertaining to the handling, analysis and transport of PCB-containing materials.
- Store all test kit components at 4 degrees Celsius (°C) to 8°C (39 degrees Fahrenheit (°F) to 46°F) when not in use. Storage at ambient temperature (18°C to 27°C or 64°F to 81°F) on the day of use is acceptable.
- Do not freeze test kit components or expose them to temperatures greater than 37°C (99°F).
- Allow all reagents to reach ambient temperature (18°C to 27°C or 64°F to 81°F) before beginning the test. This typically requires at least 30 minutes to warm from recommended storage conditions.
- Do not use test kit components after the expiration date.
- Do not use reagents or test tubes from one test kit with reagents or test tubes from a different test kit.
- Use approved methodologies to confirm any positive results.
- Distribution of PCB in soils may be highly variable and can be minimized through use of a composite sampling technique. Adequate sample number and distribution are the responsibility of the analyst.

Materials Provided

- 20 PCB antibody-coated, 12 mm X 75 mm polystyrene test tubes
- 15 milliliter (mL) Assay Diluent
- 0.5 mL Negative Control (Methanol)
- 5.0 mL PCB-Enzyme Conjugate
- 15 mL Chromogenic Substrate
- 15 mL Stop Solution
- 20-Place test tube rack
- 22 Pre-assembled 1–25 µL Gilson Microman® positive displacement pipette tips.
- 4 PCB positive assay calibrators:

  0.5 mL 1.0 ppm calibrator
  0.5 mL 5.0 ppm calibrator
  0.5 mL 10 ppm calibrator
  0.5 mL 50 ppm calibrator

NOTE: The PCB positive assay calibrators reflect the actual PCB (Aroclor) concentrations provided. See "Calibrator Concentration" for the actual PCB concentrations.

Materials You Supply

See "Ordering Information" for the appropriate catalogue numbers. To order refer to the "Technical Assistance" section for the phone numbers of the nearest Millipore office.

Methanol

Methanol (60 mL for 12 samples) is required for soil extractions.

EnviroGard Soil Extraction Kit

Use this kit for the extraction of PCB from soil samples. This kit contains the following items to test 12 samples:

- 12 Polypropylene bottles with screw caps, 30 mL (each bottle contains five stainless steel mixing beads)
- 12 Filtration devices, comprised of 12 upper (filter unit) and lower (sample tube) units
- 15 Wooden spatulas
- 12 Screw top glass storage vials, 4 mL
- 15 Weigh boats

EnviroGard Soil Field Lab
(Starter Accessory Kit)

This kit contains the following items:

- 1 Positive displacement precision pipettor, adjustable (2-250 microliters [µL])
- 1 Eppendorf™ Repeater® pipettor
- 1 Electronic timer
- 13 Polystyrene test tubes, 12 mm X 75 mm (for blanking the spectrophotometer and dilutions)
- 1 Portable balance with a 100 gram (g) calibrator weight
- 1 Wash bottle, 500 mL
- 4 Six-position test tube racks
- 100 1–25 µL positive displacement pipette tips (yellow), not pre-assembled
- 100 50–250 µL positive displacement pipette tips (pink), non-preassembled
- 8 5.0 mL pipette tips for the Repeater pipettor, (for 0.1 mL and 0.5 mL dispensing volumes).
- 4 12.5 mL pipette tips for the Repeater pipettor, (for 0.25 mL and mL dispensing volumes).
- 1 50 mL pipette tip for the Repeater pipettor (for 1.0 mL and 5.0 mL dispensing volumes).

NOTE: Order replacement pipettors and tips separately. See the "Ordering Information" section.

Millipore Differential Photometer

The Millipore Differential Photometer allows you to measure results in the form of optical density values. These values can be used for objective record keeping, quality assurance, or quantitative determination of sample concentrations from an Aroclor standard curve. See "Ordering Information" for the catalogue number.

Other

- tap or distilled water for test tubes washes

Materials Suggested but Not Required

- protective clothing (e.g., latex gloves)
- absorbent paper for blotting test tubes
- liquid and solid waste containers

Assay Procedure

Collect/Store the Sample

The following steps explain how to properly collect and store your samples.

1. Collect soil in appropriately-sized and labeled containers.

   NOTE: Take care to remove excess twigs, organic matter, and rocks or pebbles from the soil sample to be tested.

2. Soils obtained from areas adjacent to standing water, surface soils collected during or immediately after rain or snow, or any soils with relatively high amounts of water (≥ 30% by weight) should be dried before testing.

   NOTE: Contact technical service for recommended methods.

3. Store soil samples at 4°C (39°F) or room temperature *for up to 1 month*. Recommended soil storage for EPA method 8080 (gas chromatography [GC] analysis of PCBs in soil) is at 4°C (39°F).

Prepare the Sample/Extract the Soil

Refer to the *EnviroGard Field Soil Extraction* product insert.

The following steps explain how to weigh your samples using a portable balance.

1. Use the portable balance, wooden spatula, and a weigh boat to measure out 5.0 g of soil:
2. Place the balance on a level surface and press ON/MEMORY.
3. Place the weigh boat on the balance and press TARE.
4. Weigh the soil.
5. Transfer the 5.0 g of soil into a labeled, 30 mL polypropylene bottle.

NOTE: If you are testing more than one soil sample, cap the vial loosely and repeat steps 1 and 2 until all soil samples are weighed out. Use a clean weigh boat for each sample.

5. Position the Repeater pipettor at Setting 5 and use a **50 mL** pipette tip to pipette 5.0 mL of Methanol into each soil sample.

7. Cap all vials tightly and shake vigorously for approximately two minutes. Let the contents settle briefly.

8. Pour the liquid contents of each bottle into the labeled, lower (sample tube) piece of the filter base unit. To obtain optimal filtering efficiency, do not let more than one or two mixing beads slip into the filter device.

NOTE: When extracting clay samples, it is possible that the sample will soak up all of the methanol, leaving little or no excess liquid to decant. You should add an additional 5.0 mL of methanol to the sample and shake vigorously for an additional 1–2 minutes. Continue on to step 9. Make sure to factor the dilution into the calculations. See the "Results Interpretation" section.

9. Insert the plunger into the filter base unit.

10. Push down on the plunger. After 30–60 seconds, push down on the plunger again.

11. For longer term or spill-safe storage, remove the cap from the plunger and carefully pour the sample extract into an appropriately-labeled 4.0 mL glass storage vial and cap. Repeat this step for each of the sample extracts.

Perform the Test

The PCB Test Kit can be performed in either of the two following ranges:

Standard Protocol	High Sensitivity Protocol
For PCB analyses in the 5.0-50 ppm range, use a 5.0 microliter(µL) volume sample and the appropriate calibrators (5, 10, 50 ppm).	For PCB analyses in the 1.0-10 ppm range, use a 25 µL volume sample and the appropriate calibrators (1, 5, 10 ppm).

NOTE: Allow all test kit components to come to ambient temperature before use.

Follow the appropriate steps and calibrators for your protocol.

1. Label the 12 mm X 75 mm test tubes (no more than 20 tubes/assay).*

Standard Protocol		High Sensitivity Protocol	
Tube Label	**Tube Contents**	**Tube Label**	**Tube Contents**
NC	Negative Control	NC	Negative Control
5 ppm	5 ppm PCB calibrator	1 ppm	1 ppm PCB calibrator
10 ppm	10 ppm PCB calibrator	5 ppm	5 ppm PCB calibrator
50 ppm	50 ppm PCB calibrator	10 ppm	10 ppm PCB calibrator
S1	sample 1	S1	sample 1
S2	sample 2	S2	sample 2

* You do not have to perform the assay in duplicate; however, doing so increases the precision of the test.

iroGard Soil Field Lab
(Starter Accessory Kit)

This kit contains the following items:

- 1 Positive displacement precision pipettor, adjustable (2-250 microliters [µL])
- 1 Eppendorf™ Repeater® pipettor
- 1 Electronic timer
- 13 Polystyrene test tubes, 12 mm X 75 mm (for blanking the spectrophotometer and dilutions)
- 1 Portable balance with a 100 gram (g) calibrator weight
- 1 Wash bottle, 500 mL
- 4 Six-position test tube racks
- 100 1–25 µL positive displacement pipette tips (yellow), not pre-assembled
- 100 50–250 µL positive displacement pipette tips (pink), non-preassembled
- 8 5.0 mL pipette tips for the Repeater pipettor, (for 0.1 mL and 0.5 mL dispensing volumes).
- 4 12.5 mL pipette tips for the Repeater pipettor. (for 0.25 mL and mL dispensing volumes).
- 1 50 mL pipette tip for the Repeater pipettor (for 1.0 mL and 5.0 mL dispensing volumes).

NOTE: Order replacement pipettors and tips separately. See the "Ordering Information" section.

Millipore Differential Photometer

The Millipore Differential Photometer allows you to measure results in the form of optical density values. These values can be used for objective record keeping, quality assurance, or quantitative determination of sample concentrations from an Aroclor standard curve. See "Ordering Information" for the catalogue number.

Other

- tap or distilled water for test tubes washes

Materials Suggested but Not Required

- protective clothing (e.g., latex gloves)
- absorbent paper for blotting test tubes
- liquid and solid waste containers

Assay Procedure

Collect/Store the Sample

The following steps explain how to properly collect and store your samples.

1. Collect soil in appropriately-sized and labeled containers.

   NOTE: Take care to remove excess twigs, organic matter, and rocks or pebbles from the soil sample to be tested.

2. Soils obtained from areas adjacent to standing water, surface soils collected during or immediately after rain or snow, or any soils with relatively high amounts of water (≥ 30% by weight) should be dried before testing.

   NOTE: Contact technical service for recommended methods.

3. Store soil samples at 4°C (39°F) or room temperature for up to 1 month. Recommended soil storage for EPA method 8080 (gas chromatography [GC] analysis of PCBs in soil) is at 4°C (39°F).

Prepare the Sample/Extract the Soil

Refer to the *EnviroGard Field Soil Extraction* product insert.

The following steps explain how to weigh your samples using a portable balance.

1. Use the portable balance, wooden spatula, and a weigh boat to measure out 5.0 g of soil:
2. Place the balance on a level surface and press ON/MEMORY.
3. Place the weigh boat on the balance and press TARE.
4. Weigh the soil.
5. Transfer the 5.0 g of soil into a labeled, 30 mL polypropylene bottle.

NOTE: If you are testing more than one soil sample, cap the vial loosely and repeat steps 1 and 2 until all soil samples are weighed out. Use a clean weigh boat for each sample.

5. Position the Repeater pipettor at Setting 5 and use a 50 mL pipette tip to pipette 5.0 mL of Methanol into each soil sample.

7. Cap all vials tightly and shake vigorously for approximately two minutes. Let the contents settle briefly.

8. Pour the liquid contents of each bottle into the labeled, lower (sample tube) piece of the filter base unit. To obtain optimal filtering efficiency, do not let more than one or two mixing beads slip into the filter device.

NOTE: When extracting clay samples, it is possible that the sample will soak up all of the methanol, leaving little or no excess liquid to decant. You should add an additional 5.0 mL of methanol to the sample and shake vigorously for an additional 1–2 minutes. Continue on to step 9. Make sure to factor the dilution into the calculations. See the "Results Interpretation" section.

9. Insert the plunger into the filter base unit.

10. Push down on the plunger. After 30–60 seconds, push down on the plunger again.

11. For longer term or spill-safe storage, remove the cap from the plunger and carefully pour the sample extract into an appropriately-labeled 4.0 mL glass storage vial and cap. Repeat this step for each of the sample extracts.

Perform the Test

The PCB Test Kit can be performed in either of the two following ranges:

Standard Protocol	High Sensitivity Protocol
For PCB analyses in the 5.0-50 ppm range, use a 5.0 microliter(µL) volume sample and the appropriate calibrators (5, 10, 50 ppm).	For PCB analyses in the 1.0-10 ppm range, use a 25 µL volume sample and the appropriate calibrators (1, 5, 10 ppm).

NOTE: Allow all test kit components to come to ambient temperature before use.

Follow the appropriate steps and calibrators for your protocol.

1. Label the 12 mm X 75 mm test tubes (no more than 20 tubes/assay).*

Standard Protocol		High Sensitivity Protocol	
Tube Label	**Tube Contents**	**Tube Label**	**Tube Contents**
NC	Negative Control	NC	Negative Control
5 ppm	5 ppm PCB calibrator	1 ppm	1 ppm PCB calibrator
10 ppm	10 ppm PCB calibrator	5 ppm	5 ppm PCB calibrator
50 ppm	50 ppm PCB calibrator	10 ppm	10 ppm PCB calibrator
S1	sample 1	S1	sample 1
S2	sample 2	S2	sample 2

* You do not have to perform the assay in duplicate; however, doing so increases the precision of the test.

NOTE: The negative control is an optional control for assay quality control purposes.

2. Place the test tubes in the test tube rack pressing down firmly on each tube so that they are secured

CAUTION: Do not "snap" the test tubes into the rack as this may result in a cracked tube.

3. Position the Repeater pipettor at Setting **2** and use the **12.5 mL** syringe to add **500** µL of Assay Diluent to all test tubes.

4. Adjust the positive displacement pipettor dial to **050** and use the 1.0–25 µL yellow pipettor tips.

5. Use the positive displacement pipettor, to add the Negative Control (methanol) and the appropriate calibrator to the corresponding test tubes as follows:

Standard Protocol		High Sensitivity	
Calibrator	**Volume Added**	**Calibrator**	**Volume Added**
neg. control	5 µL	neg. control	25 µL
5 ppm	5 µL	1 ppm	25 µL
10 ppm	5 µL	5 ppm	25 µL
50 ppm	5 µL	10 ppm	25 µL

CAUTION: Replace the cap(s) on the calibrator vials immediately after use to minimize evaporation.

6. Briefly shake the test tube rack to mix, then incubate for 15 minutes.

7. Vigorously shake out the test tube contents into a sink or suitable container. Fill the test tubes to overflowing with cool tap or distilled water, then decant and vigorously shake out the remaining water.

8. Repeat this wash step three more times, being certain to shake out as much water as possible on each wash. After the final wash, remove as much water as possible by tapping the inverted tubes on absorbent paper.

9. Position the Repeater pipettor at Setting **2** and use the **5 mL** syringe to add 200 µL of the PCB enzyme-conjugate to all test tubes. Briefly shake the test tube rack to mix, then incubate for 5 minutes.

10. Vigorously shake out the test tube contents into a sink or suitable container. Fill the test tubes to overflowing with cool tap or distilled water, then decant and vigorously shake out the remaining water.

11. Repeat this wash step three more times, being certain to shake out as much water as possible on each wash. After the final wash, remove as much water as possible by tapping the inverted tubes on absorbent paper.

12. Position the Repeater pipettor at Setting **2** and use a clean **12.5 mL** syringe to add 500 µL of Substrate to all test tubes. Briefly shake the test tube rack to mix, then incubate for 5 minutes.

13. Position the Repeater pipettor at Setting **2** and use a **12.5 mL** syringe to add 500 µL of Stop Solution to all test tubes.

▲ WARNING: Stop solution is 1.0 N hydrochloric acid. Handle carefully.

14. Add 1.0 mL of Stop Solution to the blank test tube and insert the tube into the left well of the spectrophotometer. Dry the outside of each assay tube and measure the absorbance by placing each tube into the right well of the spectrophotometer. Record the absorbance of each tube.

NOTE: For more details refer to the Millipore Differential Photometer instructions (P17500). See the "References" section of this insert.

Results Interpretation

- Samples with OD_{450} values > OD_{450} of the 1.0 ppm PCB calibrator contain *less* than 1.0 ppm PCB.

  Samples with OD_{450} values ≤ OD_{450} of the 1.0 ppm PCB calibrator may contain *more* than 1.0 ppm PCB.

- Samples with OD_{450} values > OD_{450} of the 5 ppm calibrator contain *less* than 5 ppm PCB.

  Samples with OD_{450} values ≤ OD_{450} of the 5 ppm calibrator *may* contain *more* than 5 ppm PCB.

- Samples with OD_{450} values > OD_{450} of the 10 ppm calibrator contain *less* than 10 ppm PCB.

  Samples with OD_{450} values ≤ OD_{450} of the 10 ppm calibrator *may* contain *more* than 10 ppm PCB.

- Samples with OD_{450} values > OD_{450} of the 50 ppm calibrator contain *less* than 50 ppm PCB.

  Samples with OD_{450} values ≤ OD_{450} of the 50 ppm calibrator *may* contain *more* than 50 ppm PCB.

Soil samples that were extracted with more than 1.0 mL of methanol per gram of soil (e.g., for clay samples) require a correction factor to interpret the results. Multiply each of the calibrator concentrations by the ratio of methanol (mL) to soil (grams).

Example

If you use 10 mL of methanol to extract 5.0 g of soil, then the ratio of methanol to soil is "2" (10/5). The calibrator levels used for this soil would change to 10 ppm, 20 ppm, and 100 ppm (2 X 5 ppm, 10 ppm, and 50 ppm).

For Aroclors 1242, 1016, 1248, and 1254, the confidence interval for negative samples (i.e. ≤ 1 ppm, ≤ 10 ppm, and ≤ 50 ppm) exceeds 99%. For Aroclor 1260 the confidence interval is smaller, but still exceeds 95%.

It is possible to analyze other Aroclors not previously described in this insert, including Aroclors 1221, 1232, 1262, and 1268. Sensitivities and confidence intervals may be different for each of these. Any such analysis would require calibration with the matching Aroclor.

For more information, refer to the section, "Technical Assistance" for the number of the Millipore office nearest you.

Performance Characteristics

Sensitivity

The sensitivity is sufficient to perform the test at each calibrator level with 99% confidence. The minimum reliable detection limit for the EnviroGard PCB Test Kit is 3.3 ppm in soil for the standard protocol and 0.5 ppm in soil for the high sensitivity protocol. This is the lowest concentration of PCB in soil that is differentiated 99% of the time from zero. The sensitivity of the assay also depends on the specific Aroclor that is measured. Continue on to the "Specificity" section.

Specificity

The PCB antibody in this kit binds to different Aroclors with different affinities. The test specificity is restricted to PCBs. The test response to Aroclors 1016, 1242, 1254, and 1260 is within twofold of the response for Aroclor 1248. The calibrator levels are adjusted to detect the specified Aroclors with 95% confidence that there will be no false negatives.

Interfering Substances

The following substances were tested and found to have less than 0.5% weight-to-weight of the immunoreactivity of Aroclor 1248.

1,2-dichlorobenzene	2,5-dichlorophenol
1,3-dichlorobenzene	2,4,5-trichlorophenol
1,4-dichlorobenzene	2,4,6-trichlorophenol
1,2,4-trichlorobenzene	biphenyl
2,4-dichlorophenol	pentachlorophenol (PCP)

Limitations of the Procedure

The EnviroGard PCB Test Kit is a screening test *only*. Actual quantitation of PCBs by EnviroGard immunoassay is only possible if the contaminating Aroclor is known and if the assay is standardized using the corresponding PCB mixture.

Soil sampling error may significantly affect testing reliability. The distribution of PCBs in different soils can be extremely heterogeneous. You should homogenize soils thoroughly before analysis by any method. Split samples (e.g., for GC and immunoassay) should always come from the same homogenate.

To ensure accurate and reliable results, you should make every effort to perform the EnviroGard PCB Test at temperatures between 15°C (59°F) and 30°C (86°F).

Expected Values for PCB- Contaminated Soils

Contaminated soils have PCB levels that correlate well (correlation coefficient [r] ~ 0.9) with GC values. The slope of the correlation will depend on the contaminating Aroclor. Aroclor 1248-contaminated samples have a slope close to "1" since the EnviroGard PCB Test Kit is standardized using Aroclor 1248.

CAUTION: There is a sample size difference between the standard (5.0, 10, 50 ppm) and high sensitivity (1.0, 5.0, 10 ppm) PCB assay ranges. This differs only in the volume of calibrator or sample extract added at the beginning of the assay.

Use 5 µL when working with calibrators in the 5.0-50 ppm range. You can't achieve a 1.0 ppm sensitivity with this protocol. Use 25 µL when working with calibrators in the 1.0-10 ppm range.

If you work at 5.0 and 10 ppm action levels, use either a 5.0 mL or 25 mL volume, however, be sure to use the same sample size for calibrators and samples.

Calibrator Concentrations

Standard	Actual Concentration
1.0 ppm calibrator	0.5 ppm Aroclor 1248
5 ppm calibrator	3.0 ppm Aroclor 1248
10 ppm calibrator	5 ppm Aroclor 1248
50 ppm calibrator	22.0 ppm Aroclor 1248

Quality Control

If a blue color does not develop in the negative control test tube within 5 minutes after you add the substrate solution, the test is invalid and you must repeat the entire test.

References

Data related to the EnviroGard PCB Test Kit is on file at Millipore Corporation. Refer to the section, "Technical Assistance," for the phone number of the nearest Millipore office.

Ordering Information

The following table lists descriptions and catalogue numbers for various EnviroGard PCB and soil extraction test kits and related products.

Description	Catalogue Number
EnviroGard PCB Test Kit includes: ■ 20 PCB antibody-coated, 12 mm X 75 mm polystyrene test tubes ■ 15 mL Assay Diluent ■ 0.5 mL Negative Control (Methanol) ■ 0.5 mL 1.0 ppm PCB Calibrator ■ 0.5 mL 5 ppm PCB Calibrator ■ 0.5 mL 10 ppm PCB Calibrator ■ 0.5 mL ppm PCB Calibrator ■ 4.8 mL PCB-Enzyme Conjugate ■ 15 mL Chromogenic Substrate ■ 15 mL Stop Solution (1.0 N Hydrochloric acid) ■ 20-Place test tube rack	ENVR 000 09
EnviroGard Field Soil Extraction Kit includes the following items to test 12 samples: ■ 12 Polypropylene bottles with screw caps, 30 mL, each containing 5 stainless steel mixing beads ■ 12 Filtration devices, comprised of 12 upper (filter unit) and lower (sample tube) units ■ 15 Wooden spatulas ■ 12 Screw-top glass vials, 4 mL ■ 15 Weigh boats	ENSP 000 20
EnviroGard PCB in Soil Test Kit, shipping kit includes: ■ EnviroGard PCB Test Kit (ENVR 000 09) ■ EnviroGard Field Soil Extraction Kit (ENSP 000 20) ■ Methanol, 100 mL (ELCR 000 07)	ENVR 000 10
Methanol for soil extraction, 100 mL bottle	ELCR 000 07
EnviroGard PCB Soil Lab. Starter Accessory Kit for use with the EnviroGard PCB in Soil Test Kit, includes: ■ 1 Positive displacement precision pipettor, adjustable (2-250 µL) ■ 1 Eppendorf Repeater pipettor ■ 1 Electronic timer	ENVR L00 09

Continued

Description	Catalogue Number
■ 13 Polystyrene test tubes, 12 mm X 75 mm (for blanking the spectrophotometer and dilutions)	
■ 1 Portable balance with a 100 gram calibrator weight	
■ 1 Wash bottle, 500 mL	
■ 4 Test tube racks, six-position	
■ 100 1–25 µL positive displacement pipette tips (yellow), not pre-assembled	
■ 10050-250 µL positive displacement pipette tips (pink), non-preassembled	
■ 8 5.0 mL pipette tips for the Repeater pipettor, (for 0.1 mL and 0.5 mL dispensing volumes)	
■ 4 12.5 mL pipette tips for the Repeater pipettor, (for 0.25 mL and 0.625 mL dispensing volumes)	
■ 1 50 mL pipette tip for the Repeater pipettor (for 1.0 mL and 5.0 mL dispensing volumes)	
Millipore Differential Photometer:	
■ 115 volt (V)	ENVR 000 00
■ 230 V	ENVR 002 30
EnviroGard Replacement Pipettor Tips (available separately):	
■ Positive displacement pipettor tips, 1.0-25 mL, 200 pack, not preassembled	ENVR L04 09
■ Positive displacement pipettor tips, 50-250 mL, 200 pack, not preassembled	ENVR L07 09
■ Repeater pipettor tips, 5.0 mL, 100/pk	ENVR L01 09
■ Repeater pipettor tips, 12.5 mL, 100/pk	ENVR L02 09
■ Repeater pipettor tips, 50 mL, 10/pk	ENVR L03 09
EnviroGard PCB Test Kit (without PCB calibrators)	ENVR 0NC 09

Technical Assistance

For additional information about Millipore products, call the Millipore office nearest you.

Millipore Overseas Offices

Australia
A•C•N: 001-239-818
Toll Free: (008) 222-111
In Sydney Area:
(02) 428-7333

Austria, Central Europe, C.I.S., Africa, Middle-East, and Gulf
In Austria: (43) 1-877-8926

Baltic Republics
In Finland: (90) 801 90 77

Belgium and Luxembourg
Tel. (02) 242-17-40

Brazil
Tel. (011) 548-7011

Canada
Toll Free: 1-800-268-4881
In Toronto Area:
416-678-2161

China, People's Republic of
Beijing: (86) 1-5135114
Guangzhou: (86) 20-686217
Shanghai: (86) 21-3203856

Czech Republic
Tel. (42) 2-35-02-27
(42) 2-35-23-75

Denmark
Tel. 46-59-00-23

Finland
Tel. (90) 801-90-77

France
Tel. (1) 30-12-70-00

Germany
Tel. (06196) 494-0

Hong Kong
Tel. (852) 803-9111

Hungary
Tel. (36) 11-66-86-74

India
Bangalore:
Tel. (812) 394657

Italy
Milano: (02) 25078-1
Roma: (06) 5203600
Padova: (049) 8803720

Japan
Tel. (03) 3474-9111

Korea
Tel. (82-2) 5548305

Malaysia
Tel. (60) 3-7571322

Mexico
Tel. (525) 576-96-88

The Netherlands
Tel. (01608) 22000

Norway
Tel. 472-267-82-53

Poland
Tel. (48) 2/669 12 25/
(48) 2/663 70 31

Puerto Rico
Tel. (809) 747-8444

Singapore
Tel. (65) 253-2733

Spain
Madrid: 91-729-03-00
Barcelona: 93-325-96-16
Sevilla: 95-425-68-77

Sweden
Sundbyberg: 08-628-69-60

Switzerland
Tel. (01) 945-3242

Taiwan
Tel. (886-2) 7001742

United Kingdom and Ireland
Tel. (0923) 816375

United States of America
Tel. Toll Free
800-MILLIPORE
(800-645-5476)
In Western States, Alaska & Hawaii:
800-632-2708
In Puerto Rico:
(809) 747-8444

In All Other Countries:
Millipore Intertech
397 Williams Street
Marlborough, MA
01752-9162 U.S.A.
Tel. (508) 624-8622

General Limited Warranty

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MILLIPORE

EnviroGard™ DDT Soil Test Kit

Intended Use

The EnviroGard DDT Soil Test Kit is a qualitative or semi-quantitative field test for the detection of DDT and its metabolites *DDD* and *DDE* in soil. With the EnviroGard DDT Soil Test Kit, you can read test results visually or perform a semi-quantitative analysis using a photometer.

Test Principles

The EnviroGard DDT Soil Test Kit is based on the use of polyclonal antibodies that bind either DDT or DDT-Enzyme Conjugate. These antibodies are immobilized to the walls of the test tubes. When DDT is present in the sample, it competes with the *DDT*-Enzyme Conjugate for a limited number of antibody binding sites.

- A sample containing DDT is added to a test tube containing Assay Diluent. DDT-Enzyme Conjugate is then added to the test tube. The DDT-Enzyme Conjugate competes with the DDT for the antibody binding sites.
- After the incubation, the unbound molecules are washed away.
- A clear solution of chromogenic Substrate is then added to the test tube. In the presence of bound DDT-Enzyme Conjugate, the clear Substrate is converted to a blue color. One enzyme molecule can convert many Substrate molecules.

Since there are the same number of antibody binding sites on every test tube and each test tube receives the same number of DDT-Enzyme Conjugate molecules, a sample that contains a low concentration of DDT allows the antibody to bind many DDT-Enzyme Conjugate molecules.

Therefore, a low concentration of DDT produces a dark blue solution. Conversely, a high concentration of DDT allows fewer DDT-Enzyme Conjugate molecules to be bound by the antibodies, resulting in a lighter blue solution.

Note: Color is inversely proportional to DDT concentration.

Darker color = Lower concentration
Lighter color = Higher concentration

Performance Characteristics

The EnviroGard DDT in Soil Test Kit will not differentiate between DDT, its metabolites, and other structurally similar compounds, but will detect their presence to differing degrees. The following table shows a number of compounds and the approximate concentration of each required to yield a positive result (Lower Limit of Detection or LLD) and the concentration required to inhibit one-half of the color developed by the Negative Control (IC50). Concentration is in parts per million (ppm) in soil.

Compound	LLD	IC50
p,p'-DDT (kit calibrator)	0.04	1.25
p,p'-DDD	0.01	0.3
p,p'-DDE	0.18	3.6
o,p'-DDT	4	93
o,p'-DDD	0.4	11
o,p'-DDE	3	93
DDA	0.002	0.04
Chloropropylate	0.007	0.08
Chlorobenzilate	0.03	0.35
Dicofol	0.14	2
Tetradifon	1.2	14
Thiobencarb	5	52
Tebuconazole	7	95
Neburon	17	284
Chloroxuron	24	216
Monolinuron	25	714
Diclofop	70	>1000

The following compounds have lower limits of detection > 100 ppm:

2,4-D	4-chlorophenoxyacetic acid
Chlorbromuron	Chlordane
Chlortoluron	Dicamba
Diflubenzuron	Diuron
Lindane	Linuron
MCPA acid	MCPB
Mecoprop	

Precautions

- Treat DDT, solutions that contain DDT and potentially contaminated soil samples as hazardous materials.
- Where appropriate, use gloves, proper protective clothing, and methods to contain and handle hazardous material.
- Store all test kit components at 4°C to 8°C (39°F to 46°F) when not in use.
- Do not freeze test kit components or expose them to temperatures greater than 37°C (99°F).
- Allow all reagents to reach ambient temperature (18°C to 27°C or 64°F to 81°F) before beginning the test.
- Do not use test kit components after the expiration date.
- Do not use reagents or test tubes from one test kit with reagents or test tubes from a different test kit.
- Use approved methodologies to confirm any positive results.
- Do not dilute or adulterate test reagents or use samples not called for in the test procedure; this may give inaccurate results.
- Tightly recap the DDT calibrator vials to prevent evaporative loss.
- Distribution of DDT in soils may be highly variable. The use of a composite sampling technique may be appropriate. Development of a sampling plan that assures adequate sample number and distribution is the responsibility of the analyst.
- DDT is light sensitive. Store soil extracts at 2°C to 7°C, shielded from direct light.

Materials Provided

EnviroGard DDT Soil Test Kit
This test kit contains the following items:

20 Antibody-Coated Test Tubes
1 vial of Assay Diluent
1 vial of Negative Control (methanol)
1 vial of 0.1 ppm DDT Calibrator in methanol
1 vial of 1 ppm DDT Calibrator in methanol
1 vial of 10 ppm DDT Calibrator in methanol
1 vial of DDT-Enzyme Conjugate
1 vial of Substrate
1 vial of Stop Solution
1 20-place Test Tube Rack
22 Pipette Tips (for the Gilson M-25 Microman® Positive Displacement Pipettor)

Materials Required and Ordered Separately

See "Ordering Information" for the appropriate catalogue numbers.

EnviroGard Soil Extraction Kit

Use this kit for the extraction of DDT in soil samples. This kit contains enough devices to process 12 samples:

- 12 30 milliliter (mL) Polypropylene Bottles with screw caps (each bottle contains five stainless steel mixing beads)
- 12 Filtration Devices, comprised of 12 upper (filter unit) and lower (sample tube) units
- 15 Wooden Spatulas
- 12 Screw Top Glass Vials, 4.0 mL
- 15 Weigh Boats

Methanol

Methanol, ACS reagent grade (60 mL for 12 samples), is required for soil extraction, but is not included in the EnviroGard Soil Extraction Kit. You must order it separately. (See "Ordering Information.")

Materials Required but Not Provided

You will also need several other items, some of which are included in the EnviroGard Soil Field Lab. (See "Ordering Information" for the appropriate catalogue number).

- Gilson M-25 Microman Positive Displacement Pipettor
- Eppendorf™ Repeater® Pipetter and five Combitips® (3 x 12.5 mL, 1 x 5.0 mL, and 1 x 50 mL)
- Balance capable of accurately weighing 5 grams
- Millipore Differential Photometer or Enviro-Quant Photometer
- Indelible marker for labeling test tubes
- Watch or timer
- Clean running water or a wash bottle containing tap or deionized water (500 mL)
- Calculator (optional)

Suggestions for Pipetter Use

- Practice using both pipetters (positive displacement and Repeater pipetter) with water and extra tips before you analyze your samples.
- Use a new tip each time you use the Repeater pipetter to avoid reagent cross-contamination. Label three 12.5 mL tips "Diluent", "Substrate" and "Stop," and one 5.0 mL tip "Conjugate".
- Draw the desired reagent volume into the Repeater pipetter and dispense one portion of the reagent back into the container to properly engage the ratchet mechanism. If you do not do this, the first volume delivered may be inaccurate.
- To add reagents using the Repeater pipetter, pipette down the side of the test tube just below the rim.
- To add samples and calibrators using the positive displacement pipetter, pipette down the side of the test tube just above the liquid level.
- The carryover volume of the positive displacement tips is minimal, but may affect results if you are going from a high to low DDT concentration. Use a new pipetter tip each time you pipette a new unknown.

Assay Procedure

Collect/Store the Sample

1. Collect soil in appropriately-sized and labeled containers.
2. Take care to remove excess twigs, organic matter and rocks or pebbles from the sample. For best results, wet soils should be air-dried overnight and thoroughly mixed before testing.
3. Store soil samples at 4°C (39°F).

Prepare the Sample/Extract the Soil

1. Use the portable balance, wooden spatula, and a weigh boat to measure out 5.0 g of soil.
   - Place the balance on a level surface and press ON/MEMORY.
   - Place the weigh boat on the balance and press TARE.
   - Weigh the soil.
2. Transfer the 5.0 g of soil into an appropriately-labeled, 30 mL polypropyiene extraction bottle. If you are testing more than one soil sample, cap the bottle loosely and repeat steps 1 and 2 until all soil samples are weighed out. Use a new wooden spatula and weigh boat for each sample.
3. Attach a 50 mL Combitip to the Repeater pipetter and set the dial to 5. Add 5.0 mL of methanol to the extraction vial and cap tightly.
4. Repeat steps 1-3 for each sample you test.
5. Vigorously shake the extraction bottles for at least 2 minutes and then allow the contents to settle briefly.
6. Pour the liquid contents of each vial into the appropriately-labeled lower (sample tube) piece of the filter unit. To obtain optimal filtering efficiency, do not let more than one or two mixing beads slip into the filter device.
7. Insert the plunger into the filter base unit.
8. Push down on the plunger to filter the extract. After 30 to 60 seconds, push down on the plunger again.
9. For longer term or spill-safe storage, remove the cap from the plunger and carefully pour the sample extract into an appropriately-labeled, 4.0 mL glass vial. Cap the vial. Repeat this step for each of the sample extracts.
10. If sample extracts are not going to be tested right away, store them at 2°C to 7 °C in the dark.

Perform the Test

NOTE: Allow all reagents and sample extracts to reach room temperature before you begin the test. Do not analyze more than 20 test tubes at a time.

1. The choice of calibrators to use in the test will depend on the the selection of the analysis. The use of two calibrators may be appropriate if screening for a single level of DDT.

Remove the test tubes from the plastic bag and label them as follows*:

Tube Label	Tube Contents
NC	Negative Control
C1	0.1 ppm Calibrator
C2	1 ppm Calibrator
C3	10 ppm Calibrator
S1	sample 1
S2	sample 2
etc.	

* You are not required to perform the assay in duplicate; however, doing so will increase the precision.

Place the test tubes in the test tube rack. Push down on each tube so that it is held firmly and does not fall out of the rack when shaken.

CAUTION: Do not "snap" the test tubes into the rack as this may result in a cracked tube.

2. Attach the 12.5 mL Combitip labeled "Diluent" to the Repeater pipetter and adjust the dial to 2. Add 500 microliters (µL) of Assay Diluent to each test tube.
3. Attach a clean pipette tip to the Microman pipetter and adjust the dial to "250". Add 25 µL of each calibrator to the corresponding "C" test tube by placing the end of the pipette tip against the side of the tube (just above the level of the Assay Diluent) and dispensing the volume.

CAUTION: Replace the caps on the calibrator vials immediately after use to minimize evaporation.

4. Using a clean tip for each sample, add 25 µL of each sample extract to the appropriately-labeled test tube.
5. Attach the 5.0 mL Combitip labeled "Conjugate" to the Repeater pipetter and adjust the dial to 1. Add 100 µL of DDT-Enzyme Conjugate to each test tube.
6. Shake the test tube rack to mix for 10 to 15 seconds. Leave the test tubes undisturbed for 15 minutes.
7. Vigorously shake out the test tube contents into a sink or suitable container. Fill the test tubes to overflowing with cool tap or distilled water, then decant and vigorously shake out the remaining water.

   Repeat this wash step three more times, being certain to shake out as much water as possible on each wash. After the final wash, remove as much water as possible by tapping the inverted tubes on absorbant paper.
8. Attach the 12.5 mL Combitip labeled "Substrate" to the Repeater pipetter and set the dial to 2. Add 500 µL of Substrate to each test tube. Leave the test tubes undisturbed for 10 minutes.

   NOTE: If a blue color does not develop in the Negative Control test tube within 10 minutes after adding the Substrate, the test is invalid and you must repeat it.

Interpret the Results

You can either interpret the results visually within 10 minutes after adding the Substrate to each test tube, or you can perform a more precise analysis with a photometer after you add the Stop Solution.

Visual Interpretation

After you add the Substrate, wait 10 minutes then mix the test tubes by shaking them for a few seconds until they are a uniform blue color. Compare the sample test tube to the calibrator test tubes against a white background. The test tube rack in the kit is well-suited for this purpose.

NOTE: The word DDT in the interpretation instructions below refers to "total DDT", i.e. the sum of *p,p'*-DDT, *p,p'*-DDD, and *p,p'*-DDE.

- If a sample test tube contains *more* color than the calibrator test tube, the sample contains DDT at a concentration *lower* than the calibrator.
- If a sample test tube contains *less* color than the calibrator test tube, the sample may contain DDT at a concentration *greater* than the calibrator.
- If the sample test tube contains color that is between the calibrator test tubes, the sample contains DDT at

a concentration between the calibrator concentrations.

- If a sample test tube contains *approximately the same* amount of color as the calibrator test tube, the sample contains DDT at a concentration *approximately equal* to the calibrator.

- If the sample test tube contains less color than the 10 ppm Calibrator test tube, you may dilute a fraction of the soil extract in methanol (for example, 1:100) and perform the assay again. To determine the concentration of the diluted extract multiply the result by the dilution factor. (Go to "Semi-Quantitative Interpretation" for further details.)

Photometric Interpretation

After you add the Substrate, wait 10 minutes then add the Stop Solution to each test tube.

WARNING: Stop solution is 1N Hydrochloric acid. Handle carefully.

Attach the 12.5 mL Combitip labeled "Stop" to the Repeater pipetter and set the dial to 2. Add 500 µL of Stop Solution to each test tube. This converts the blue color in the test tubes to yellow.

NOTE: After you add Stop Solution to the test tubes, results should be read within 30 minutes.

Millipore Differential Photometer

1. Place a water blank test tube containing 1.5 mL of Milli-RO® or Milli-Q® water, or equivalent in the left (reference) well.

2. Place the Negative Control test tube into the right (sample) well. Record the optical density (OD) of the Negative Control.

3. Remove the Negative Control test tube and replace it with the 0.1 ppm Calibrator test tube to reactivate the photometer. Record the result. Repeat this step to determine the OD for each of the remaining calibrators and for each sample.

Semi-quantitative Interpretation

Compare the OD of each sample to the OD of each calibrator:

NOTE: The word DDT in the interpretation instructions below refers to "total DDT", i.e. the sum of *p,p'*-DDT, *p,p'*-DDD, and *p,p'*-DDE.

- If a sample OD is *equal* to the OD of a calibrator, the sample contains DDT at a concentration *approximately equal* to the calibrator.

- If a sample OD is *greater* than a calibrator OD, the sample contains *less* DDT than the calibrator.

- If a sample OD is *lower* than a calibrator OD, the sample may contain *more* DDT than that calibrator.

- If an assay result indicates that a soil sample contains greater than 10 ppm total DDT, but you need more specific information, the soil extract may be diluted 1:100 in neat methanol, and assayed again. You must then multiply the results of the re-assay by 100 to determine the approximate sample concentration.

NOTE: If you know in advance that the "action level" of interest is greater than 10 ppm total DDT in soil, the assay may be modified to pinpoint that particular concentration. For example:

If you wish to categorize samples as less than or greater than 250 ppm, you should dilute all sample extracts 1:250 in neat methanol (e.g. 20 µL extract plus 4.98 mL methanol) and compare the diluted extracts to the 1 ppm DDT kit calibrator. Due to the 250-fold dilution, the 1 ppm calibrator represents 250 ppm in the assay.

NOTE: If you are interested in action levels greater than 1000 ppm, please contact Millipore Technical Services for assistance.

Limitations of the Procedure

The EnviroGard DDT Soil Test Kit is a screening test only. Actual quantitation of DDT by EnviroGard immunoassay is not possible due to the Test kit's cross-reactivity with DDT breakdown products and other similar compounds and to the variations in extraction efficiency inherent in the fast extraction protocol described in this product insert.

Soil sampling error may significantly affect testing reliability. The distribution of pesticides in different soils can be extremely heterogeneous. Soils should be dried and homogenized before analysis by any method. Split samples (i.e. for GC and immunoassay) should always derive from the same homogenate.

Example

Actual OD values will vary. This data is for demonstration purposes only.

Tube	OD	Interpretation
NC	0.90	
C1 (0.1 ppm)	0.75	
C2 (1 ppm)	0.49	
C3 (10 ppm)	0.35	
S1	0.68	>0.1 ppm < 1 ppm
S2	0.16	> 10 ppm

Note:

The EnviroQuant Photometer is also available from Millipore. This dual wavelength instrument measures the OD at 450 nm minus 600 nm of all samples and calibrators, and provides a printout of results. See "Ordering Information" for the appropriate catalogue number.

Ordering Information

The following table lists descriptions and catalogue numbers for the EnviroGard DDT and Soil Extraction Test Kits and related products.

Description	Catalogue Number
EnviroGard DDT in Soil Test Kit, includes the following items: • 20 Antibody-Coated Test Tubes • 1 vial of Assay Diluent • 1 vial of Negative Control • 1 vial of 0.1 ppm DDT Calibrator • 1 vial of 1 ppm DDT Calibrator • 1 vial of 10 ppm DDT Calibrator • 1 vial of DDT-Enzyme Conjugate • 1 vial of Substrate • 1 vial of Stop Solution (1N hydrochloric acid) • 1 20 - place Test Tube Rack • 22 Pipette Tips, 1 to 25 µL range (for the Gilson M-25 Microman positive displacement pipettor)	
EnviroGard Field Soil Extraction Kit, includes the following items to test 12 samples: • 12 30 mL Polypropylene Bottles with screw caps, each containing 5 stainless steel mixing beads • 12 Filtration Devices, comprised of 12 upper (filter unit) and lower (sample tube) units • 15 Wooden Spatulas • 12 Screw-Top Glass Vials, 4.0 mL • 15 Weigh Boats	ENSP 000 20
Methanol for soil extraction, 100 mL bottle	ELCR 000 07
Millipore Differential Photometer: • 115 volt (V) • 230 V	 ENVR 000 00 ENVR 002 30
EnviroQuant Photometer, 110V EnviroQuant Photometer, 220V EnviroQuant Replacement Paper, 12 rolls	ENVR T11 00 ENVR T22 00 ENVR T11 02
EnviroGard Replacement Pipetter Tips (available separately): • Positive displacement pipetter tips, 1-25 µL range 200/pk (not preassembled) • Repeater pipetter tips, 5.0 mL, 100/pk • Repeater pipetter tips, 12.5 mL, 100/pk • Repeater pipetter tips, 50 mL, 10/pk	 ENVR L04 09 ENVR L01 09 ENVR L02 09 ENVR L03 09

Description	Catalogue Number
EnviroGard Soil Test Kit includes: • 1 Positive displacement precision pipetter • 1 Eppendorf Repeater pipetter • 1 Electronic timer • 13 Polystyrene test tubes, 12 mm X 75 mm (for blanking the spectrophotometer and sample dilutions) • 1 Portable balance with 100 gram calibrator weight • 1 Wash bottle, 500 mL • 4 Test tube racks, six-position • 8, 5.0 mL Pipette tips for the Repeater pipetter, for 0.1 mL through 0.5 mL dispensing volumes • 4, 12.5 mL Pipette tips for the Repeater pipetter, for 0.25 mL through 1.250 mL dispensing volumes • 1, 50 mL Pipette tip for the Repeater pipetter, for 1.0 mL through 5.0 mL dispensing volumes • 100 pipette tips, 2.0-25 µL (not preassembled) • 100 pipette tips, 50-250 µL (not preassembled) Contact Millipore Technical Service for kit component replacement or reordering information. (See the "Technical Assistance" section for the number of the Millipore office nearest you.)	ENVR L00 09

Technical Assistance

For additional information about Millipore products, call the Millipore office nearest you.

Millipore Overseas Offices

Australia
A•C•N: (001) 239-818
Toll Free (008) 222-111
In Sydney Area (02) 428-7333

Austria, Central Europe, C.I.S., Africa, Middle-East and Gulf
In Austria: (43) 1-877-8926

Baltic Republics
In Finland: (90) 801 90 77

Belgium and Luxembourg
(02) 242-17-40

Brazil
Tel. (011) 548-7011

Canada
Toll Free 1-800-268-4881
In Toronto Area:
416-678-2161

China, People's Republic of
Beijing: (86) 1-5135114
Guangzhou: (86) 20-686217
Shanghai: (86) 21-3203856

Czech Republic
Tel. (42) 2-35-02-27
(42) 2-35-23-75

Denmark
Tel. (4 =9-00-23

Finland
Tel. (90) 801-90-77

France
Tel. (1) 30-12-70-00

Germany
Tel. (06196) 494-0

Hong Kong
Tel. (852) 803-9111

Hungary
Tel. (36) 11-66-86-74

India
Bangalore:
Tel. (812) 394657

Italy
Milano: (02) 25078-1
Roma: (06) 5203600
Padova: (049) 8803720

Japan
Tel. (03) 3474-9111

Korea
Tel. (82-2) 5548305

Malaysia
Tel. (60) 3-7571322

Mexico
Tel. (525) 576-96-88

The Netherlands
Tel. (01608) 22000

Norway
Tel. 472- 67-82-53

Poland
Tel. (48) 22-44-76-10
(48) 22-44-09-02

Singapore
Tel. (65) 253-2733

Spain
Madrid: 91-729-03-00
Barcelona: 93-325-96-16
Sevilla: 95-425-68-77

Sweden
Sundbyberg:
08-628-69-60

Switzerland
Tel. (01) 945-3242

Taiwan
Tel. (886-2) 7001742

United Kingdom and Ireland
Tel. (0923) 816375

United States of America
Tel. Toll Free
1-800-645-5476
In Western States, Alaska & Hawaii:
1-800-632-2708
In Puerto Rico:
(809) 747-8444

In All Other Countries:
Millipore Intertech, U.S.A.
Tel. (508) 624-8622
Fax (508) 624-8630

General Limited Warranty

Millipore Corporation ("Millipore") warrants the products manufactured by it against defects in materials and workmanship when used in accordance with the applicable instructions for a period of one year from the date of shipment of the products or where applicable, for a period not to extend beyond a product's printed expiration date. MILLIPORE MAKES NO OTHER WARRANTY, EXPRESSED OR IMPLIED. THERE IS NO WARRANTY OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE. The warranty provided herein and the data, specifications and descriptions of Millipore products appearing in Millipore's published catalogues and product literature may not be altered except by express written agreement signed by an officer of Millipore. Representations, oral or written, which are inconsistent with this warranty or such publications are not authorized and if given, should not be relied upon.

In the event of a breach of the foregoing warranty, Millipore's sole obligation shall be to repair or replace, at its option, any product or part thereof that proves defective in materials or workmanship within the warranty period, provided the customer notifies Millipore promptly of any such defect. The exclusive remedy provided herein shall not be deemed to have failed of its essential purpose so long as Millipore is willing to repair or replace any nonconforming Millipore product or part. Millipore shall not be liable for consequential, incidental, special or any other indirect damages resulting from economic loss or property damage sustained by a customer from the use of its products.

However, in some states the purchaser may have rights under state law in addition to those provided by this warranty.

Safety

To receive complete safety information on this product, contact the nearest Millipore office and request Material Safety Data Sheet documents P70002, P70003, P34207 and P34210.

Other

This kit was developed in collaboration with the Commonwealth Scientific and Industrial Research Organization (Australia) using reagents produced and supplied under exclusive license to Millipore and ImmunoSystems Incorporated.

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